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ELEMENTARY PRACTICAL CHEMISTRY

AND

QUALITATIVE ANALYSIS

BY

PROFESSORS CLOWES AND COLEMAN.

PRACTICAL CHEMISTRY AND QUALITATIVE ANALYSIS; specially adapted for Colleges and Schools. By FRANK CLOWES, D.Sc., Emeritus Professor of Chemistry in University College, Nottingham. Seventh Edition, with 101 Engravings, 576 pages, Post 8vo, 8s. 6d.

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ELEMENTARY PRACTICAL CHEMISTRY

AND

QUALITATIVE ANALYSIS

BY

FRANK CLOWES, D.Sc. Lond.

EMERITUS PROFESSOR OF CHEMISTRY IN THE UNIVERSITY COLLEGE, NOTTINGHAM
MEMBER OF COUNCIL OF THE CHEMICAL SOCIETY
VICE-PRESIDENT OF THE INSTITUTE OF CHEMISTRY
PAST-PRESIDENT OF THE SOCIETY OF CHEMICAL INDUSTRY
FELLOW OF THE GERMAN CHEMICAL SOCIETY
CHEMICAL ADVISER TO THE LONDON COUNTY COUNCIL

AND

J. BERNARD COLEMAN, A.R.C.Sc.

PROFESSOR OF CHEMISTRY IN THE SOUTH-WESTERN POLYTECHNIC, CHELSEA, S.W.
FELLOW OF THE INSTITUTE OF CHEMISTRY AND OF THE CHEMICAL SOCIETY

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PREFACE.

The Text-Book on Qualitative Analysis, written by one of us, has proved by its success that it meets the requirements of large numbers of teachers and students. It furnishes a fairly complete course of work for a student who intends to make a study of Qualitative Analysis.

We have long felt, however, that a smaller treatise would be useful to students of elementary practical chemistry and chemical analysis, who have the opportunity of working in a suitably equipped laboratory and under the direction of a teacher. The course of instruction in this book is adapted to those students who do not need so thorough a chemical training as that which must be undertaken by the future professional and analytical chemist. This smaller book is therefore intended for the general students and the technical students of our schools and colleges.

The book is divided into two parts, the first being devoted to General Chemistry, the second to Analytical Chemistry.

Part I. comprises a course of instruction, which gradually presents the principles of chemistry to the student, by means of a selected series of experiments.

This part commences with a description of the metric system of weights and measures. After this, concise directions are given for the preparation and use of some simple bench-apparatus. The student next studies the composition of air and water, then the process of combustion and the properties of some of the more important non-metals and their compounds. Many quantitative experiments are given, including some which illustrate the laws of chemical combination and of chemical equivalence, and the relation of acids to alkalis as regards their neutralising power.

82551

This part concludes with a selection of examples designed to test the progress of the student.

Since it is now customary for the student in the Laboratory to work on parallel lines with the course pursued by the teacher in the lecture-room, it is anticipated that the instruction given in this portion of the book will form a suitable basis upon which a teacher may found his lecture course. Throughout this part experiments have been selected which can be readily performed by a student; and experiments which are more suited for a lecture and require considerable manipulative skill have been purposely omitted.

Part II. describes the analytical reactions of the commonly occurring metals and inorganic acid-radicles, and the methods of detecting them when they occur singly or in mixture.

In order to keep this portion of the book within small dimensions, the reactions and methods of detection of the less commonly occurring metals and acid-radicles and of organic substances have been omitted.

In order to enable the course of work to be further curtailed, if necessary, some of the less important portions of the text have been printed in smaller type. These may be passed over by a student whose time is very limited.

A glance at the Contents on pages vii–xvii will serve to indicate in greater detail the general arrangement and scope of the work.

Chemical symbols and formulæ have been freely used throughout the book. In order to assist beginners, the chemical name has always been associated with the formula when this is employed for the first time: lists of symbols and formulæ, together with their corresponding names, have also been supplied in the Appendix.

F. C.
J. B. C.

London, *March* 1900.

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APPARATUS FOR EACH BENCH.

The following is a List of the Apparatus which will be required by each student for the following course of work.

The numbers in brackets in this List refer to the paragraphs in the book in which the apparatus is described.

Other apparatus will be occasionally required, and will be used in common by the students working in the laboratory.

Full descriptions of all laboratory-apparatus and of the reagents required for Qualitative Analysis will be found in *Clowes' Qualitative Analysis*, Section VII.

1	Bunsen-burner with tubing (19).
1	Test-tube stand, with ten $\frac{3}{4}$ inch holes and two $1\frac{1}{4}$ inch.
12	Test-tubes, 5 inches by $\frac{5}{8}$ inch.
1	Test-tube brush (29).
2	Boiling-tubes, 6 inches by 1 inch.
2	Round glass plates, 3 inches across.
2	Porcelain dishes, 3 inches across.
2	Watch-glasses, 2 inches across.
1	Four-ounce conical flask.
1	Wedgwood pestle and mortar, 4 inches across.
1	Iron tripod-stand (fig. 27, page 32).
1	Piece of coarse iron-wire gauze, 5 inches square.
3	Glass funnels, 2–$2\frac{1}{2}$ inches across.
	Cut filter-papers, 3·5 and 4·5 inches in diameter.
3	Small beakers, 3 oz., 5 oz., and 7 oz. in capacity.
3	Glass rods (26).
1	Piece of platinum foil, 1 inch by $1\frac{1}{2}$ inch.
2	Pieces of mounted platinum wire (25).
1	Black's blowpipe (23).
1	Pipe-clay triangle (fig. 27, page 32).
1	Wash-bottle, 18 oz. in capacity (28).
1	Retort-stand, rod 16 inches, 3 rings (fig. 29, page 41).
1	Wooden filter-stand (fig. 44, page 73).
1	Pair of gun-metal crucible tongs, 6 inches long.
1	Ebonite spatula, 4 inches in length.
1	Wicker draining basket, 10 inches by 8 (29).

PART I.

GENERAL CHEMISTRY.

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PART I.

SECTION I.

MEASUREMENT OF LENGTH, AREA, VOLUME, AND WEIGHT.

RELATIVE DENSITY OF SOLIDS AND LIQUIDS.

It is essential that the chemical student, before commencing any quantitative measurements, should be acquainted with the measurements described below.

It may, however, be advisable, in certain cases, to perform only the exercises on length, volume, and weight at the commencement of the course, and to reserve the remainder of the exercises for a later stage.

If the measurements have already been carried out in the Physical Laboratory, they need not be repeated.

For convenience in reference, paragraph numbers are printed in the text in thick numbers, and in square brackets at the top of each page.

THE DECIMAL OR THE METRIC SYSTEM.

1. The Metric System.—The system of measurement almost universally adopted for scientific investigations is known as the 'metric' system.

The standard of length is the metre. A bar of platinum is kept in Paris, which measures precisely this length at the temperature of 0° C.

The metre was originally intended to be one 10,000,000th of that quadrant of the earth's surface, which is drawn from the North Pole to the Equator and which passes through Paris. The measurement of this quadrant was, however, not absolutely correct, and the standard metre does not therefore exactly correspond to the original definition.

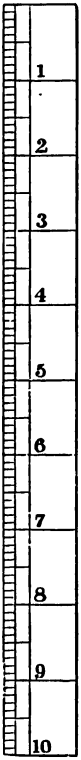
The other measures of length are derived from the metre. The larger measures are multiples by ten, and the smaller ones are sub-multiples by ten of the metre. This relation of the measures has led to the designation 'decimal.'

It will be seen that measurements taken on this system are very conveniently written down in the usual decimal numerical notation, and that a simple and direct relation exists between the unit of volume, the unit of weight, and the unit of length (4, 11).

In the English system of weights and measures, the relation between the multiples and sub-multiples of the unit is arbitrary and wanting in uniformity, and no simple connection exists between the units of length, of volume, and of weight.

The tables of weights and measures are fully set forth in the Appendix, and a reference to these tables, and to the accompanying explanatory matter, will fully illustrate the above statements.

FIG. 1.



ONE DECIMETRE.

MEASUREMENT OF LENGTH.

2. The Metre is the metric unit of length and is equal to 39·37 inches. It is divided into tenths, hundredths, and thousandths, and these divisions are named by prefixing the Latin terms *deci*, *centi*, and *milli* respectively to the word metre.

The multiples of the metre, which are respectively ten, one hundred, and one thousand times as great as the metre, are designated by prefixing the Greek numerals, *deka*, *hecto*, and *kilo* respectively to the 'metre.'

These prefixes are employed in a precisely similar sense for distinguishing the different measures of area, of capacity, and of weight (3, 4, 11).

The diagram (fig. 1) represents one decimetre sub-divided into centimetres and into millimetres.

The sub-divisions and multiples of the metre are tabulated below, and the different columns indicate the relation of these units to the metre, and to the English units; the last column states also the usual contractions of the French names.

MEASURES OF LENGTH.

		Metre.		English equivalent.	Written.
Millimetre	=	$\frac{1}{1000}$	0·001	0·03937 ins.	mm.
Centimetre	=	$\frac{1}{100}$	0·01	0·3937 ,,	cm.
Decimetre	=	$\frac{1}{10}$	0·1	3·937 ,,	dcm.
Metre	=	1	1·0	39·37 ,,	m.
Dekametre	=	10	10·0	10·936 yards.	dkm.
Hectometre	=	100	100·0	109·36 ,,	hm.
Kilometre	=	1000	1000·0	1093·61 ,,	km.

1 inch = 2·54 centimetres.

The sub-divisions of the metre which are most frequently used for chemical purposes are the centimetre and the millimetre.

Exercise 1.—Measure the length of a page of this book in inches with a foot-rule; then make the same measurements by means of a half-metre rule, in decimetres, centimetres, and millimetres.

Exercise 2.—On a sheet of ruled foolscap paper count off twenty-one lines, and measure the distance of each line from the top line in inches; make these measurements also in centimetres and millimetres.

Exercise 3.—Mark off two distances of one inch and of ten inches on paper; then measure off the former length in millimetres, and the latter length in centimetres.

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MEASUREMENT OF AREA OR SURFACE.

3. The Unit of Area in the metric system is the square metre.

Since in linear metric measure the values of the measures increase and diminish in multiples and sub-multiples of ten, it follows that in square metric measure the values will increase and diminish in multiples and sub-multiples of one hundred. Thus, since 1 decimetre is equal to 10 centimetres, it follows that 1 square decimetre will be equal to 100 square centimetres.

The sub-multiples of a square metre are:—

1 sq. cm. = 100 sq. mm.
1 sq. dcm. = 10,000 „ „
1 sq. m. = 1,000,000 „ „

Exercise 4.—Find the area of one sheet of single foolscap paper in square centimetres and square inches, remembering that the area of a rectangle is obtained by multiplying its length by its breadth.

Exercise 5.—Find the area of a penny, a circular glass plate, and a filter paper in sq. mm. and sq. dcm., remembering that the area of a plane circle is equal to the square of its radius multiplied by 3·14.

MEASURES OF VOLUME OR CAPACITY.

4. **The Standard of Volume** in the metric system is the cubic metre: and since the value of the linear measures in the metric system increase and diminish by multiples and by sub-multiples of ten, the values of the cubic measures will increase and diminish by multiples and sub-multiples of one thousand. Thus:—

1 cubic metre = 1,000 cubic decimetres (c. dcm.)
= 1,000,000 cubic centimetres (c. c.)
= 1,000,000,000 cubic millimetres (c. mm.)

MEASUREMENT OF VOLUME OF SOLIDS.

5. For chemical purposes it is seldom necessary to measure the volume of a solid. A few examples of the measurement of solids are, however, given below, in order to familiarise the student with the relative volumes denoted by the cubic inch, the cubic decimetre, and the cubic centimetre.

The volume of an irregular solid which is heavier than water can be obtained by the method described in Exercise 8, par. 7.

Exercise 6.—Find the volume of a given rectangular block of wood in cb. ins., in cb. dcms., and in c.cs. (The volume of a rectangular solid = length × breadth × depth.)

Exercise 7.—Find the volume of a cylindrical vessel in cb. ins. and in c. cs. (The volume of a cylinder = length × square of the radius × 3·14.)

MEASUREMENT OF VOLUME OF LIQUIDS.

6. The direct measurement of the volume of a liquid and of a gas is an operation which must frequently be performed by the chemist, but the volume is also often ascertained indirectly.

Thus the volume of a liquid may be calculated from its weight, if the specific gravity of the liquid is known; and the volume of a gas may be indirectly measured by ascertaining the volume of a suitable liquid which the gas can displace.

Hence the measurement of the volume of a liquid is one of the most common operations performed by the chemist.

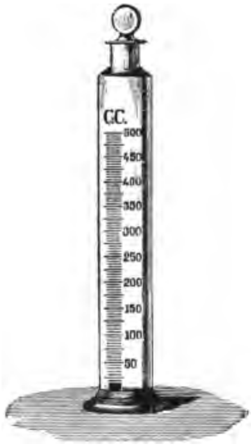
It has been found convenient to adopt a special series of units for the measurement of the smaller quantities of liquid which are commonly dealt with in the laboratory. The standard unit selected for this purpose is the cubic decimetre or *litre*, and this contains 1000 cubic centimetres. The litre, half-litre, quarter-litre, and cubic centimetre are in constant use in laboratory measurements.

The names of the multiples and sub-multiples of the litre are formed by using the same prefixes as those which have been given in connection with linear measurement (**2**). A table of the metric measures of capacity, together with their English equivalents, is given in the Appendix. The litre is equivalent to 1·76 pints.

APPARATUS FOR MEASUREMENT OF LIQUID VOLUME.

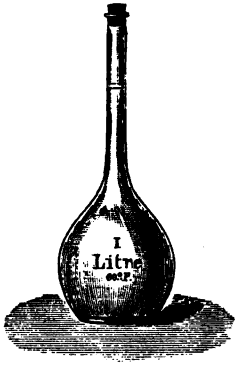
7. The Measuring Cylinder is generally used for measuring a comparatively large volume of liquid. It is a cylindrical vessel, which is graduated throughout the greater part of its length. The graduations indicate either cubic centimetres or multiples of cubic centimetres, according to the capacity of the cylinder. The top of the cylinder is either furnished with a stopper (fig. 2) or is left permanently open. When the measurement of a liquid is being taken, the level of the bottom of the curve of the liquid surface should be read off. Since the cylinder is furnished with a continuous series of graduations, it may be used for measuring either the total volume for which it is graduated, or any fraction of that volume.

Fig. 2.



The Measuring Cylinder.

Fig. 3.



The Litre Flask.

8. **The Measuring Flask** (fig. 3) is graduated for measuring only one definite volume of a liquid, and cannot be used for measuring fractions of that volume. The figure shows a flask for measuring one litre (1000 c.c.) Other convenient flasks in common use are those for measuring 500 c.c., 250 c.c., and 100 c.c.

The upper surface of the liquid, during measurement, is in the neck of the flask, and the lowest point of the curve (see fig. 7, p. 9) should just touch the etched mark.

In fig. 3 two marks are shown. When the flask is filled up to the lower mark it contains exactly the volume stated; when it is filled up to the higher mark, the stated volume may be poured out, or *delivered*, from the flask.

Exercise 8.—Ascertain *by displacement* the volume of a solid of irregular shape, such as a glass stopper, by filling a measuring cylinder to the 50 c.c. mark with water, then entirely immersing the stopper, and noting the increase of volume registered by the surface of the liquid.

Exercise 9.—Ascertain by measurement the volume of a regularly-shaped piece of metal, and compare this volume with that obtained by displacement (Exercise 8).

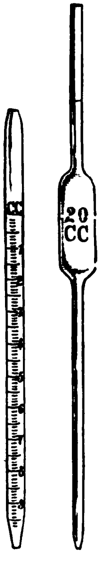
9. The Pipette is used for delivering a small measured volume of a liquid. It usually consists of a tube narrowed at both ends.

Two kinds of pipette are in use. One of these has only one mark (fig. 5), and serves to deliver only one definite volume of liquid. The other (fig. 4) is graduated throughout the greater part of its length, and serves to deliver either its maximum volume or a fraction of that volume.

In using the pipette, the liquid is sucked up past the graduation mark, and is retained by placing the first finger quickly over the upper end. The pressure of the finger is then slightly relaxed, and the liquid is allowed to flow out until the *lower curve* of its surface (fig. 7, page 9) just reaches the graduation; the finger is then firmly pressed down again so as to arrest the flow.

When the liquid is to be 'delivered' from the pipette, the finger is removed, or its pressure is relaxed, until the liquid has flowed out, and the pipette is then allowed to drain for a few seconds in a vertical position so as to deliver the liquid which adheres to its inside.

Figs. 4, 5.



Pipettes.

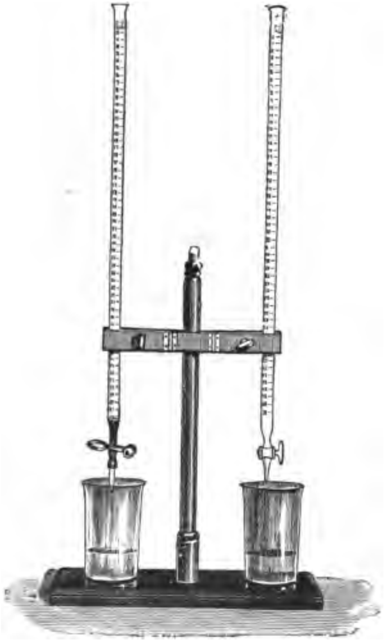
Exercise 10.—Ascertain whether the graduations of the measuring cylinder correspond with those of the pipette, by filling the 10, 25, and 50 c.c. pipettes successively with water to their marks and emptying them into the graduated cylinder. The volume of liquid which is read off within the cylinder should correspond exactly with the volume which has been added from each of the pipettes.

Enter the results in the note-book in the manner shown in the following example:—

Volume delivered from the pipette.	Volume read off in the cylinder.	Error.
10 c.c.	10 c.c.	0·0 c.c.
35 c.c.	34 c.c.	−1·0 c.c.
85 c.c.	86 c.c.	+1·0 c.c.

10. **The Burette** is used when it is necessary to measure moderate volumes of liquid with great accuracy. Two forms of burette are shown in fig. 6. They consist of long tubes open at the top and narrowed at the bottom. One is closed by a glass stopcock, and the other by means of a piece of rubber tubing and a pinchcock.

Fig. 6.



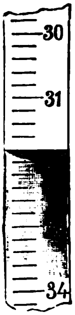
Burettes.

The burette is graduated to measure c.cs., and each c.c. is subdivided by graduations into tenths or fifths. The most convenient capacities for burettes are 25 c.c. and 50 c.c.

The burette is completely filled with liquid from the top, after the lower end has been closed. A little of the liquid is then allowed to flow out through the tap or rubber tube in order to sweep out air-bubbles. The liquid is then allowed to flow out gradually, until the curved under surface, called the meniscus, just reaches the zero graduation near the top of the burette. The appearance of the meniscus is shown in fig. 7, and the volume of liquid shown in this figure would be read as 32 c.c.

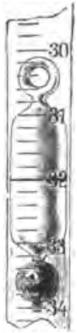
When the burette is to be used, the volume of liquid which it contains is read off, the stopcock is then opened, and as soon as the requisite quantity of liquid has flowed out, the volume is again read off. The difference between the two readings gives the volume of the liquid which has flowed from the burette.

FIG. 7.



THE MENISCUS.

FIG. 8.



ERDMANN'S FLOAT.

A light glass float known as Erdmann's float (fig. 8) is sometimes used to facilitate the reading of the liquid volume. The position of the horizontal line on the centre of the float is read off on the burette.

The accuracy of the measuring cylinder may be tested for every 25 c.c. of its capacity by means of the burette. Each of these spaces should contain exactly 25 c.c. of liquid measured from the burette.

The accuracy of the pipette may be tested by closing it at its lower end by a stretched piece of rubber-band, the ends of which are fastened by thin copper wire round the pipette. The recently drained pipette is then carefully filled to the mark from the burette, and should register the same volume of liquid as that which has flowed from the burette.

Exercise II.—Test the accuracy of the graduation of the measuring cylinders and of the pipettes by filling them to their graduations by the corresponding volume from the burette. The interior of the cylinders must be dried. The pipettes must be filled and then emptied and drained for a few seconds before the water is introduced from the burette. This difference of treatment is necessary because the cylinder is required to measure the volume of liquid which it *contains*, the pipette to *deliver* the volume of liquid denoted at the graduation.

Enter the results in columns as is shown in the table on page 8.

MEASUREMENT OF MASS.

11. **The Quantity of Matter in a Substance or its Mass** is obtained by comparing its mass with the mass of suitable standards. These standards are usually called weights, and the operation of ascertaining mass is called weighing.

The Unit of Mass in the metric system is the gram, and this is defined as *the mass of one cubic centimetre of water at 4° C.*

The simple relation which exists in the metric system between the mass of a substance and its length and volume, lessens and simplifies calculations so considerably that multiples and sub-multiples of the gram are used by the scientific chemist to the exclusion of their English equivalents.

The following table shows the metric weights :—

TABLE OF DECIMAL WEIGHTS.

	Gram.	Written.
Milligram . .	= 0·001	mgrm.
Centigram . .	= 0·01	cgrm.
Decigram . .	= 0·1	dcgrm.
Gram . . .	= 1·0	grm.
Dekagram . .	= 10·0	dkgrm.
Hectogram . .	= 100·0	hgrm.
Kilogram . .	= 1000·0	kgrm.

The weights which are most frequently used by the chemist are the gram and the kilogram.

THE BALANCE AND WEIGHTS.

12. **The Balance**, which is shown in fig. 9, is sufficiently accurate for the experiments described in this book.

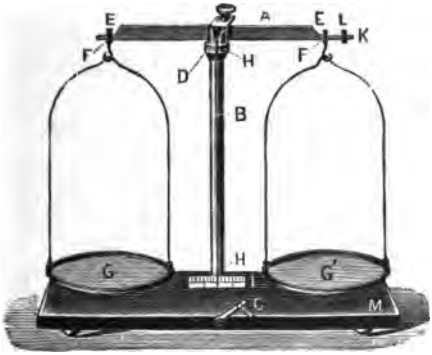
The beam (A) works upon agate knife-edges (D), and the pan hooks (F) are also suspended on knife-edges (E). By means of the lever (C), shown in front of the figure, the beam can be lifted bodily from the supporting pillar (B):

the wear of the knife-edges, when the balance is not in use, is thus prevented.

The pans (GG′) are about 4 inches in diameter, and are removable.

A pointer (HH), swinging in the front of a scale (I), serves to indicate when the pans are in equilibrium. This will be the case when the pointer oscillates through equal distances on each side of the zero mark of the scale. If the pans of the balance are not found to be in equilibrium when they are empty, equilibrium may be attained by turning the nut (L), which is seen to the right hand of the beam, in the necessary direction on the screw-thread (K). The wooden base (M) of the balance should rest on a steady table.

Fig. 9.



The Balance.

13. The Weights.—A box of weights ranging from 100 grams to 1 centigram will be sufficient for the experiments described in this book. Since these weights are either multiples or submultiples of ten, the weight of a substance is most conveniently written down in the decimal notation.

Thus a weight of 10 grams, 4 decigrams, 5 centigrams would be written 10·45 grams.

The weights as supplied by different makers are differently marked. Thus the fractions of the gram which have been first named may be marked in any of the ways shown below.

0·4 gram = 4 decigrams = 40 centigrams = 400 milligrams.
·05 ,, = 0·5 ,, = 5 ,, = 50 ,, .

Usually, however, they are marked either in decimal fractions of a gram or in milligrams. A little practice renders their use easy.

If the balance is not likely to be disturbed, the weights may be conveniently placed upon a piece of stiff cardboard, ruled off into as many squares as there are weights (fig. 10). Each of these spaces is somewhat larger than the weight which is to be placed upon it, and the value of the weight is written on the square. Accordingly, when the weight is removed to the scale pan, its value may be at once read off from the square which is left uncovered.

Fig. 10.

50	20	20	10
5	2	2	1
·5	·2	·2	·1
·05	·02	·02	·01

Card for Weights.

Exercise 12.—Remove all the weights from the box. Draw in your note-book as many squares as there are weights, and mark them with the value of the weights as is shown in fig. 10. Mark the weights less than a gram in fractions of a gram and in milligrams.

14.—The Process of Weighing.—The following rules should be observed when a substance is being weighed :—

(a.) Bring the beam to rest by means of the lever before a substance or weight is placed upon the pan of the balance.

(b.) Place the substance which is to be weighed upon the left-hand pan of the balance and the weights upon the right-hand pan.

(c.) Do not handle the weights with the fingers, but lift them by means of the brass forceps.

(d.) Place the weights upon the pan in a systematic order. Commence with a weight which is heavier than the substance to be weighed, and substitute for this the lighter weights in succession until the right one is found. Pursue a similar plan with the supplementary weights. This method secures an equipoise more quickly and certainly than a random selection of weights would do.

(e.) The substance may be considered to be balanced by the weights when the pointer swings through equal distances successively on both sides of the central mark of the graduated scale.

(f.) Unless the substance is a solid of fair size, and will not injure the material of the balance, it must not be placed directly upon the scale-pan.

(g.) Most substances can be weighed upon a watch-glass or in a small dish, which has been previously balanced, either by weights, or by small shot or pieces of lead or tinfoil.

(h.) A substance should be weighed while its temperature is the same as that of the room; if it is much colder or much hotter than the surrounding air, the body will appear heavier or lighter than it should do.

(i.) A non-volatile liquid is weighed in a balanced beaker or dish; a volatile liquid is weighed in a stoppered bottle.

Exercise 13.—Place weights amounting to 60 grams upon the left-hand scale-pan, balance these by means of the other lower weights, and see if the weights on the right-hand pan add up exactly to 60 grams.

Repeat the exercise by placing 6 grams and 0·6 grams respectively upon the left-hand pan, and counterpoising them as before.

Exercise 14.—Ascertain the weight of three coins or pieces of metal of which the weight is known; check and record the results in your note-book.

RELATION OF WEIGHT TO VOLUME IN THE METRIC SYSTEM.

15. It has been stated in paragraph 11 that the unit of weight is the mass of one cubic centimetre of water at 4° C. This simple relationship between volume and weight in the case of water affords a ready means of ascertaining the capacity of vessels in which water can be weighed. It is only necessary for this purpose to fill the previously weighed or balanced vessel to the necessary point with water, at the temperature of 4° C., and to ascertain the weight in grams of the water which has been introduced. The number of grams of water will give the number of c.c. in its volume.

Since water kept in the laboratory will usually have a temperature of about 15°·5 C., it is more convenient to use water at this temperature than at 4° C. for the above purpose; for, without involving serious error, it may be considered that the volume occupied by 1 gram of water at 15°·5 C. is identical with the volume of the same weight at 4° C. In fact most measuring vessels are actually graduated by making this assumption and by using water at 15°·5 C.

A few exercises on the comparison of the weight of water with its volume are appended.

Exercise 15.—Measure out 10 c.c. of distilled water from a pipette into a weighed beaker, and ascertain the increase of weight of the beaker. The number of grams should correspond to the number of c.c. Repeat the above process with the 20 c.c. and with the 50 c.c. pipettes.

Exercise 16.—Allow 10 c.c., 20 c.c., and 30 c.c. of distilled water successively to flow from a burette into a weighed beaker. Weigh the beaker after each addition of water, and compare the weights and volumes respectively as before. These experiments, if they are carefully carried out, will serve to check the accuracy of the burette.

Exercise 17.—Calibrate a flask to hold 75 c.c. as follows. Weigh a flask about 80 c.c. in capacity, and add 75 grammes to the weights on the pan. Then introduce distilled water carefully into the flask until the balance is restored. The level of the upper surface of the liquid should now be in the neck of the flask. Scratch a line on the neck with a wetted file or with a diamond pencil just on a level with the lowest point in the under surface of the liquid. This will give the point to which the flask must be filled to hold 75 c.c. at the temperature of the water used.

Exercise 18.—Test the accuracy of the 100 c.c. measuring flask by ascertaining the weight of water which it contains when it is filled precisely to the mark.

RELATIVE DENSITY, OR SPECIFIC GRAVITY.

16. It will have been noticed that portions of different substances, which are of equal size, possess very different weights. A cubic inch of lead is four times as heavy as a cubic inch of glass, and eleven times as heavy as a cubic inch of water.

The Relative Density or Specific Gravity of a substance is the ratio between the weight of a certain volume of the substance and the weight of an equal volume of a standard substance. Since the standard for solid and liquid substances is water, and in the above example lead is stated to be eleven times as heavy as an equal volume of water, the specific gravity of lead is 11.

The specific gravity of a substance can therefore be ascertained by dividing the weight of the substance by the weight of an equal volume of water.

Water at 4° C. is the standard of specific gravity; but it is found more convenient for ordinary purposes to employ water at the average temperature of the laboratory, which is 15°·5 C.

17. Specific Gravity of a Solid Heavier than Water.—

When a heavy solid is immersed in water, it displaces its own volume of water; it also apparently loses a part of its weight, and this is equal to the weight of the water displaced. Hence the specific gravity of such a solid may be found by dividing the weight of the solid taken in the air by the loss of weight which it suffers by complete immersion in water.

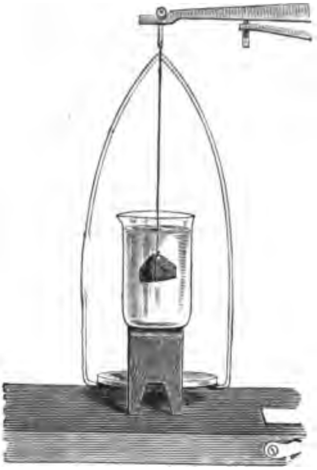
If W = the weight of the solid in the air
and W_1 = the weight of the solid in water,

$$\text{then the Spec. Grav. of the solid} = \frac{W}{W - W_1}.$$

In applying this method the solid substance is first suspended, by means of a fine silk thread, about midway between the beam of the balance and the scale-pan, and it is then weighed.

A beaker nearly full of distilled water at 15°·5 C. is then brought up under the substance until the substance is totally immersed in the liquid, the beaker being supported on a wooden bridge, which is placed over the left hand pan in such a way as not to impede its movement (fig. 11). Care must be taken that any air-bubbles which adhere to the immersed solid are brushed off, and that the solid does not touch the sides or bottom of the beaker.

FIG. 11.



WEIGHING A SOLID IN WATER.

The substance is then again weighed while it is totally immersed in the water, and its specific gravity is calculated as is described above.

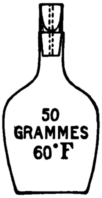
Exercise 19. — Ascertain the specific gravity of a solid glass stopper, of a piece of calc-spar, and of a piece of lead in the manner described above.

18. Specific Gravity of a Liquid.

—The usual method for determining the specific gravity of a liquid consists in first finding the weight of water at 15°·5 C. which a suitable bottle of known weight will hold. The bottle is then filled with the liquid of which the specific gravity is required, and its weight is again ascertained. The specific gravity is obtained by dividing the weight of the liquid which fills the bottle by the weight of the water.

The ordinary specific gravity bottle with a perforated stopper may be used for this purpose (fig. 12).

FIG. 12.



THE SPECIFIC GRAVITY BOTTLE.

A convenient substitute may be made by removing the stopper from a one-ounce bottle, and filing a groove along its ground surface at right-angles to the base, by means of a wetted triangular file.

Clean and dry the bottle and ascertain its weight. Then fill the bottle up to the top of the neck with distilled water at 15°·5 C., and insert the stopper. The water should completely fill the bottle and the groove or perforation in the stopper.

Sometimes it is more convenient to pour the distilled water into the bottle and then to bring its temperature to 15°·5 by immersing the bottle in a vessel of water at a suitable temperature.

Now wipe the bottle dry with a clean cloth and weigh it at once. The difference between the weights of the bottle when it is empty and when it is full will give the weight of water which the bottle contains.

Empty the bottle and dry it perfectly inside and out. Then proceed to find the weight of the liquid which fills the bottle, in precisely the same manner as the weight of water was ascertained. The weights of equal volumes of water and of the liquid are now known. The specific gravity of the liquid is found by dividing the weight of the liquid by the weight of the water.

In a particular determination of the specific gravity of methylated spirit the following results were obtained :—

	Grams.
Weight of bottle, empty	**24·56**
Weight of bottle, filled with water at 15°·5 C. .	**74·54**
Weight of bottle, filled with methylated spirit at 15°·5 C.	**65·92**

Hence the specific gravity of the spirit $= \dfrac{65{\cdot}92 - 24{\cdot}56}{74{\cdot}54 - 24{\cdot}56}$

$$= \frac{41{\cdot}36}{49{\cdot}68} = 0{\cdot}827$$

Exercise 20.—Find the specific gravity of methylated spirit and of a saturated solution of common salt.

SECTION II.

PREPARATION AND USE OF APPARATUS.

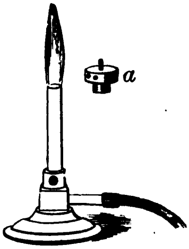
A student who is commencing work in a chemical laboratory will find, immediately before Section I., a list of the apparatus which is constantly required for working through the examples arranged in this book. This apparatus should be kept in a private locker, and should be looked through, fitted, and cleansed as is directed in this Section.

For convenience in reference, paragraph numbers are printed in the text in thick numbers, and in square brackets at the top of each page.

Paragraphs which are printed in small type may be omitted if time is limited, or they may be simply read through.

19. **The Bunsen Burner** (fig. 13) is generally employed in the laboratory for heating purposes. It is so constructed that the coal-gas, before being burnt, is mixed with a proper proportion of air, which is drawn in through holes at the lower part of the burner. The flame is thus rendered non-luminous and smaller than the bright flame. It deposits no soot upon a cool object. Its high temperature, non-luminosity, and colourless appearance also render it very valuable for producing flame colorations, as will be seen hereafter. The burner is provided with means for partly or entirely closing the air-holes when necessary. This is usually effected by turning round a loose perforated ring, which is slipped over the holes.

FIG. 13.



THE BUNSEN BURNER.

When the burner is to be used, it is connected, by means of a piece of tightly-fitting rubber tubing, about 5-16ths of an inch in internal diameter, with the tube which supplies gas to the working bench. The gas-tap is then turned on, and in a few seconds the gas is lighted. The flame should be almost perfectly colourless and should give scarcely any light.

When a small flame is used, the supply of air should be reduced in order to prevent the flame from receding and *burning below.* If the flame should recede, the gas supply must be stopped by pinching the rubber tube; the supply of air is then reduced, and the gas is again lighted.

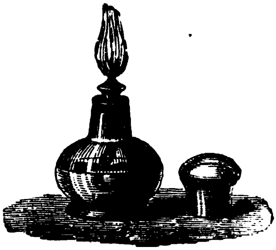
Exercise 21.—Try with flames of different sizes the effect of opening, then partially closing, and finally of entirely closing the air-holes of the burner.

When the heat of the burner is to be diffused over a larger surface, a small perforated metal cap, called a *rose-top* (fig. 13 *a*), is placed upon the top of the burner. It yields a circle of flames, and thus spreads the area directly affected by the flame.

20. The Spirit-lamp is occasionally employed instead of the gas burner; but for general purposes it should only replace this burner when coal-gas cannot be obtained.

The spirit-lamp (fig. 14) consists of a glass vessel containing methylated spirit, in which the lower end of a cotton wick is immersed. The wick is supported by means of a brass or stoneware wick-holder. When the lamp is not in use, the upper end of the wick should be covered with the glass cap, in order to prevent the evaporation of the spirit.

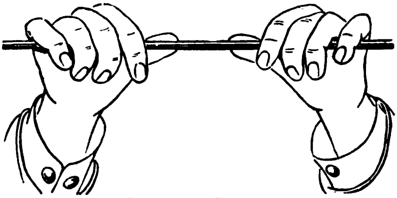
FIG. 14.



THE SPIRIT-LAMP.

21. Glass Tube or Rod is Cut by making a deep scratch with the edge of a three-cornered file across the necessary part. The glass is then held with both hands, as is shown in fig. 15, and a gentle pressure is exerted upon the glass as if trying to break it across at the scratch. If the file-mark has been made sufficiently deep, the glass will readily break at this point.

Fig. 15.

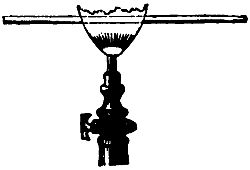


Cutting Glass.

The sharp edges of a freshly-cut rod or tube should always be rounded, either by holding them in the Bunsen-flame or blowpipe flame until they are partly melted, or by rubbing them with the face of a file.

22. Glass tube is Bent by holding it in the upper edge of a common fish-tail gas flame, as is shown in fig. 16. The tube is constantly turned slowly round on its axis, so as to heat all sides equally. As soon as the glass is felt to be soft and pliable, it is quickly bent to the required angle. The heated part must not be allowed to touch anything until it is cold. It is then cleansed from soot.

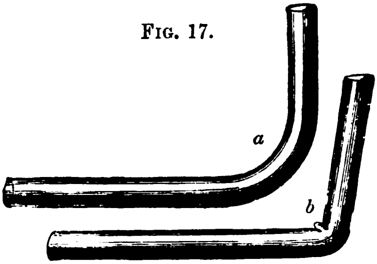
Fig. 16.



Bending Glass.

If the bend is properly made, the tube will assume the form of a curve, and its bore will not be altered (fig. 17 *a*). If the tube is bent at a sharp angle (fig. 17 *b*), its bore will be narrowed; such a bend is not only less sightly, but it will be liable to break under a small strain.

Fig. 17.



Good Bend (*a*), Bad Bend (*b*).

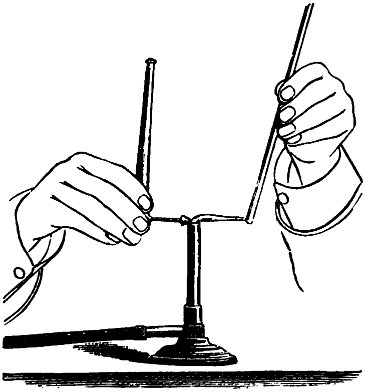
Glass rod may be bent in the Bunsen-flame or in the blowpipe flame.

Exercise 22.—Cut off a piece of glass tubing about six inches in length and one-eighth inch in bore, and bend it at a right angle in the fish-tail burner.

23. The Blowpipe is used for producing a small but hot flame, which may be made to assume any required direction. This may be effected by blowing a fine stream of air through an ordinary flame. The blowpipe is held as is shown in fig. 18: its mouth-piece is taken between the lips, and after the cheeks have been blown out to their full extent, the air contained in them is forced out through the jet. This produces a small pointed conical flame in the direction of the blast.

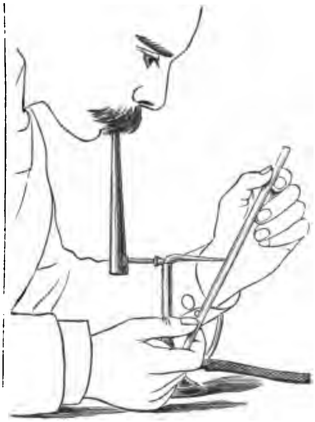
The chief difficulty which will be experienced in using the blowpipe properly arises from the necessity of maintaining the blast of air uninterrupted by the breathing. In order to effect this the cheeks must be kept inflated with air, and this air must be forced through the blowpipe *by the pressure of the cheeks alone and not by the action of the lungs.* Breathing must be carried on meanwhile through the nose, the cheeks being occasionally replenished with air from the lungs.

FIG. 18.



ROUNDING END OF GLASS ROD.

FIG. 19.



DRAWING OFF GLASS TUBE.

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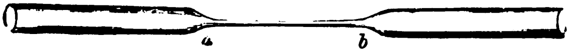
It is frequently necessary to have both hands free while the blowpipe is being used. This may be secured by resting the jet on the top of the burner and supporting the mouthpiece by the lips alone, as is shown in fig. 19.

The bright flame, obtained by closing the air-holes of the burner, is better suited for ordinary blowpipe work than the ordinary non-luminous flame.

A Herapath or Fletcher blowpipe, which is fed with air either from the foot-bellows, or from a mechanical or water-blower, is often indispensable for maintaining a high temperature, or for extensive glass-working or glass-blowing.

24. Small Ignition-tubes.—A piece of hard glass tubing may be drawn out at its middle point by heating it strongly in the Bunsen- or in the blowpipe-flame,* as is shown in fig. 19. While the tube is being heated, it should be constantly turned around upon its long axis, and as soon as the glass becomes soft, it is gradually drawn out by pulling its ends in opposite directions. It will now assume the shape shown in fig. 20.

FIG. 20.

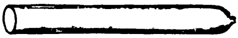


GLASS TUBE DRAWN OUT.

If the conical parts (*a*, *b*) are then successively softened in the flame, and each of the narrow tubes is drawn off, two small closed tubes will be obtained similar to that shown in fig. 21.

If the closed end of the tube is strongly heated in the flame, and is then gently blown into while it is red-hot, it may be expanded into a small bulb.

FIG. 21.



IGNITION TUBE.

Small test-tubes, three inches long by half an inch in diameter, will also serve for ignition-tubes.

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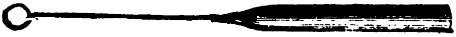
* The Bunsen-flame may be used for tube of small bore, but the flame of the foot-blowpipe will be required for thick-walled combustion-tube of larger bore.

Exercise 23.—Heat a piece of glass tube, about six inches long, in the Bunsen- or blowpipe-flame, and draw it out and form two closed tubes as has been just described. Then blow out the end of one of these tubes into a small bulb.

25. **Mounted Platinum-wire.**—A piece of platinum wire may be fixed into a glass handle in the following way.

Exercise 24.—Draw out a piece of glass tube, about five inches in length, at its middle point (figs. 19, 20), and cut it across at the middle of the narrow portion. Each piece of tube thus obtained serves for the handle of a wire. Break off the narrow part of the tube until it extends only about a quarter of an inch from the shoulder (*a*, fig. 20). Insert the end of a piece of platinum wire about three inches in length into this narrow opening, and hold the end of the tube in the blowpipe-flame until the glass melts and thickens around the wire, and fixes it firmly when it is cold (fig. 22). Then roll the free end of the wire round a stout wire, so as to form a loop of the same size and shape as is shown in fig. 22.

FIG. 22.



MOUNTED PLATINUM WIRE.

26. **Glass Stirring-rods** are required for stirring liquids. They should be cut from a piece of solid glass rod of such lengths that they will extend slightly beyond the edges of the porcelain-dish, the beakers and the small flask, when they are placed in these vessels. After they have been cut, their sharp edges should be carefully rounded off (21).

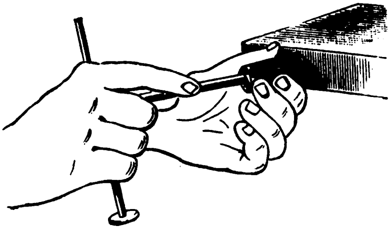
Exercise 25.—Cut some glass rod (21) into lengths of three, six, and seven inches. Heat both ends of each of these rods to redness in the Bunsen- or blowpipe-flame, turning the rod meanwhile constantly round upon its long axis (fig. 18). The sharp edges are thus removed. The end of the rod must not be allowed to touch anything until it is cool.

Heat part of an ordinary rod in the Bunsen- or blowpipe-flame until it is soft (fig. 19), then draw it out to the requisite degree (fig. 20) to produce a thin rod, such as is required occasionally for special purposes.

27. **Corks are Bored** by means of the cork-borer, which is a brass or steel tube sharpened at one end.

A borer is selected of slightly less diameter than the glass tube which is to be inserted into the cork. The cork is then pressed against a wooden surface, and the perforation is made by gently pushing the borer through it with a constant movement of rotation upon its axis (fig. 23).

FIG. 23.



BORING A CORK.

A slender round file is used for smoothing the interior of the hole made by the cork-borer, or for slightly enlarging it. Great care must be taken to leave the hole round in shape, and not to enlarge it so much that the glass tubing fits in too loosely.

The cork-borer is sharpened by rubbing the outer part of its edge obliquely with the face of a fine-toothed file.

In Perforating Rubber Stoppers the borer should be sharp, and its diameter should be slightly greater than that of the hole which is required. The borer should be lubricated by wetting it with water.

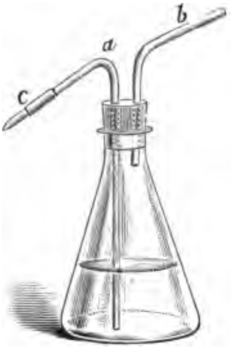
28. **The Wash-bottle.**—The fitting of the wash-bottle will serve as a good illustrative exercise on the preceding operations.

Exercise 26.—Select a thin flat-bottomed conical flask of about 18 ounces capacity, and with a neck about an inch in diameter (fig. 24); also a sound cork which is slightly too large to enter the neck of the flask. Roll the cork backwards and forwards under the foot with gentle pressure, so as to soften it and make it fit tightly into the neck of the flask.

Then bend two pieces of glass tubing (**22**), rather longer than would be required for the tubes (*a*, *b*), into the form shown in fig. 24. Cut off their ends to the right length, and round off the sharp edges (**21**).

Now make two parallel holes in the cork by means of a cork-borer (**27**). These holes must be somewhat smaller than the glass tubes, and must be smoothed, and slightly enlarged if necessary, by means of the round file, until the tubes (*a*, *b*) enter somewhat stiffly, when they are pushed into them.

FIG. 24.



THE WASH-BOTTLE.

A Rubber Stopper is much more durable than a cork for this and for most other chemical purposes. It may be purchased with two holes already made, or may be perforated by a sharp wetted cork-borer (fig. 23). Both the glass tubes and the inside of the holes should be well wetted before the tubes are inserted, since water serves as a lubricant for glass against rubber.

Before the fitting of the flask is proceeded with, it should be ascertained that all the fittings are air-tight. With this object in view, insert the cork with the tubes into the neck of the flask. Close one tube with the finger, and blow down the other tube. If any leakage of air occurs, it is as a rule easily heard; or if the outside of the cork is wetted, the escape of air-bubbles will become visible.

If the cork is air-tight, fit upon the tube (*a*) a piece of rubber tubing about an inch in length. Into the other end of this rubber tube push a short jet (*c*), made by drawing out a piece of glass tubing in the flame (fig. 19, p. 21).

The fitting of the flask is now complete, but its neck may be bound round with twine like the handle of a cricket bat, or tightly covered with a folded strip of flannel, in order to render it possible to hold the neck when it is hot.

The wash-bottle is now nearly filled with distilled water, and is ready for use.

Tap-water should never he kept in the wash-bottle.

A fine stream of water may be obtained from the jet (*c*) by blowing down the tube (*b*). This stream serves for washing precipitates and for many other purposes.

If a larger stream is required, the flask is inverted. The water will then flow out from the end of the broad tube (*b*), and air will enter meanwhile by the narrow jet tube (*a*).

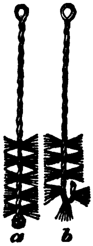
When hot water is required for washing purposes, the wash-bottle is placed upon a piece of coarse iron-wire gauze supported by a tripod-stand, and is heated by the Bunsen-flame.

29. Cleaning Apparatus.—It is indispensable to the success of analytical and of most chemical operations that all the glass and porcelain apparatus which is used should be scrupulously clean, and before beginning work the student will do well to clean his apparatus as is directed below.

Test-tube Brush.—This brush is constantly used for cleaning glass and porcelain apparatus. Two forms are shown in fig. 25.

Test-tubes, Beakers, and Porcelain dishes are washed in a stream of tap-water. If this fails to remove the stain, the surface may be wetted with hot dilute hydrochloric acid, or even with strong sulphuric or nitric acid.* Hot caustic potash solution may be required to remove grease. Speaking generally, when a substance is to be removed from the surface of a vessel to which it strongly adheres, it should be treated by a liquid in which it is easily soluble.

FIG. 25.



TEST-TUBE CLEANER.

Apparatus should be washed as soon as possible after it has been used, since the surface is usually cleansed with greater difficulty after standing.

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* *Caution.*—The student should bear in mind that acids are corrosive liquids, and that if they are spilt upon any surface they must be *at once* washed off. They will rapidly stain and destroy the clothes, and should be neutralised on the clothes *at once* by solution of ammonium hydrate or of ammonium carbonate.

The following directions in small type apply more especially to apparatus which is used in analytical operations.

Test-tubes containing liquids are supported in the test-tube stand, and after they have been washed they should be placed mouth downwards in the wicker basket to drain.

Glass Funnels should have their narrow stems cut off to within about an inch from the shoulder. The sharp outer edge must then be removed by rubbing it with the face of the triangular file.

Platinum Foil and Wire are cleansed by boiling them in hydrochloric acid and rinsing off the acid with water. The wire should then be strongly heated for some time in the blowpipe-flame. This treatment must be repeated until the wire no longer persistently colours the flame, when it is wetted with pure strong hydrochloric acid and held in the Bunsen-flame. If the tip of the wire cannot be cleansed in this way, it should be cut off. Commercial platinum is sometimes alloyed with barium or with some other metal which colours the flame. Wire made from such platinum is useless for flame coloration tests.

Instead of cleaning platinum foil and wire immediately before they are used, it is better to keep them in a small beaker containing moderately strong hydrochloric acid. When the platinum is removed from the acid and rinsed with water, it will then usually be found to be clean and not to colour the flame.

Before Apparatus is put away, all glass and porcelain which is not in actual use should be washed, and the smaller apparatus should be placed in a wicker basket to drain. The basket is then put away with its contents. *Dirty* apparatus should never be kept in the basket. All iron apparatus should be carefully dried, and must be kept in a dry place to prevent it from rusting. *Metal apparatus must never be put into the wicker draining basket.*

30. Heating Porcelain and Glass.—The two following rules should be attended to when either a glass or a porcelain vessel is being heated, in order to minimise the risk of cracking it.

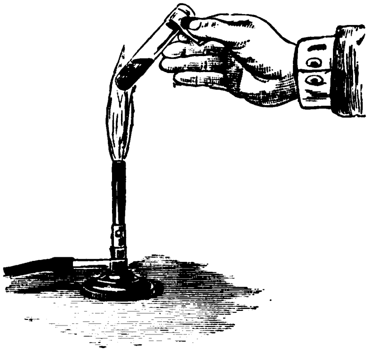
The outside of a vessel which contains a liquid should never be heated by the flame above the level of the liquid inside.

A dry hot vessel should be allowed to cool before it is placed on a cold surface, and before any liquid is poured into it.

Porcelain Dishes are generally used for the purpose of boiling or evaporating liquids. They are usually placed upon a pipeclay triangle, which is then supported by a tripod or retort-stand (fig. 27, p. 32). They may be safely heated by contact with a small naked flame.

Porcelain Crucibles are used for containing solid bodies which are to be strongly heated. They are supported in the same way as porcelain dishes. The flame should not at first be allowed to play steadily upon the bottom of the crucible so as to heat it suddenly; but the burner should be constantly moved slightly from side to side until the porcelain is hot. The crucible should be allowed to cool slowly on the triangle. The hot crucible and cover are handled by means of the crucible-tongs.

FIG. 26.



HOLDER FOR HEATING TUBE.

Glass Vessels require to be Heated more cautiously than those made of porcelain. A large naked flame must never be allowed to play for any length of time on one part of the glass surface. When a test-tube or boiling-tube is being heated, this *local heating* is prevented by holding the tube obliquely with its lower part in the flame, and either moving it gently up and down, or constantly turning it round on its axis.

Small quantities of liquid may be boiled in a test-tube, or in a boiling tube. The risk of burning the fingers by steam is avoided by bending round the neck of the tube a strip of folded *glazed* paper, or of leather, and pinching the ends of the strip together, close to the tube (fig. 26).

Beakers and glass flasks are most safely heated by placing them upon a piece of coarse wire-gauze supported by a tripod stand.

SECTION III.

CHEMICAL AND PHYSICAL CHANGE.
MIXTURES, COMPOUNDS, AND ELEMENTS.
INDESTRUCTIBILITY OF MATTER.

31. Chemical Change as distinguished from Physical Change.

Expt. 27.—Hold a piece of platinum wire, or a thread of asbestos, in the crucible tongs, and heat it in the Bunsen-flame. Note that the substance becomes red or even white hot, but that it is unaltered by being heated, and resumes its usual appearance and properties when it has cooled.

Expt. 28.—Heat a piece of bright copper wire in the same way, and notice the appearance of its surface when it has cooled. The wire will be found to have become covered with a thin grey coating.

Expt. 29.—Heat a piece of magnesium ribbon in the same way; the magnesium will burn brilliantly, and will be totally altered in appearance and properties.

In the case of the platinum and asbestos, the change was a physical change, temporary in nature. The copper, however, suffered a permanent change on its *surface*, and the magnesium was entirely and permanently altered. The two latter cases were cases of chemical change.

In all cases of chemical change the substance or substances which are formed differ in some respects from the bodies from which they have been produced. The difference is frequently in colour, in density, in solubility, or in shape.

32. Illustrations of Chemical Change.

Expt. 30.—Mix a small quantity of 'reduced iron,' or of very fine iron filings which have been sifted through muslin, with half its volume of flower of sulphur. Transfer some of the mixture to a dry test-tube and heat it gently in the Bunsen-flame. The mixture will glow, and if the product is turned out of the tube when it is cold, it will be seen to be dark grey in colour.

This is an instance of *chemical combination*. The change has been accompanied by a rise of temperature, and has caused an alteration in the colour of the substances which have combined.

Expt. 31.—Place in the bottom of a dry test-tube a small quantity of red oxide of mercury, hold the test-tube in an almost horizontal position, and heat the oxide strongly. Small globules of mercury or quicksilver will soon collect on the inside of the tube, just above the oxide.

As soon as the mercury is seen, introduce into the tube the glowing end of a slip of wood (fig. 30, p. 43). The wood will burst into flame, and will burn more brilliantly than it does in air. The kindling is due to the presence in the tube of an invisible gas called oxygen, which has been expelled from the mercury oxide.

The minute globules of mercury may be made to unite into visible drops by rubbing the glass with a glass rod or with a strip of wood.

The above experiment is an instance of chemical *decomposition*. The red mercury oxide has been decomposed into metallic mercury and the gas oxygen.

Expt. 32.—Place a small piece of copper sulphate (blue vitriol) in a test-tube, then pour in some water, and heat the water gently until the substance is dissolved. Pour most of the blue solution into a small porcelain dish, reserving a small portion in the test-tube. While the liquid in the dish is still warm, add to it half a watch-glass full of *clean* iron filings, and stir the liquid and the filings together with a glass rod.

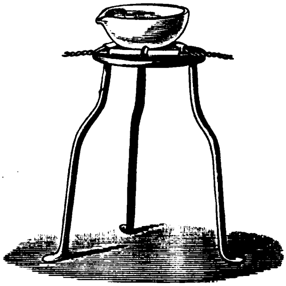
After the dish has stood at rest for a short time, red copper will be seen to have separated out, and the colour of the liquid will have changed from blue to pale green. The alteration in the properties of the blue liquid may further be shown by making the following experiments.

Place two drops of solution of potassium ferrocyanide (yellow prussiate of potash), by means of a glass rod, on separate parts of a filter-paper. Then take out a drop of the original blue solution, which was reserved in the test-tube, and place it on one of the drops on the filter-paper. A chocolate-brown stain will be produced. Place a drop of the pale green liquid from the dish on the other drop of ferrocyanide; a deep blue stain will be produced. This shows that the character of the liquid has been changed by the action of the iron filings.

This experiment is an instance of chemical *replacement*. A portion of the iron has been dissolved in the liquid, and has taken the place of the copper which has been separated from the sulphate.

Expt. 33.—Place in two small porcelain dishes about equal quantities of sodium carbonate (washing soda). Then pour some water into one dish, and notice that the solid slowly dissolves when it is stirred with the water. Pour dilute hydrochloric acid (spirits of salt) into the other dish, and note that effervescence occurs and the solid rapidly dissolves.

Fig. 27.



Evaporation in Porcelain Dish.

Now place each of the dishes successively upon a triangle on a tripod-stand, and gently heat it with a small Bunsen-flame. The liquid will gradually pass off as vapour, or *evaporate*. When the volume of the liquid becomes small, reduce the size of the flame in order to prevent the pasty mass from spirting out of the dish. After the liquid has been completely evaporated, continue to heat for a short time, so as to thoroughly dry the contents of the dish. Now compare the residues left in the two dishes. Both are white and similar in appearance; but if a little of the contents of the one which contained water is tasted, it will still be found to have the taste of washing soda, whereas the other residue will have the taste of common salt.

The water has, in fact, produced the solution or liquefaction of the carbonate of soda, and this is a change which does not permanently alter the substance; hence evaporation restores it unaltered. The acid has, however, produced a permanent or chemical change of the sodium carbonate into sodium chloride, and the sodium chloride therefore remains after evaporation and not the original sodium carbonate.

33. **Mixtures and Compounds.**—When the red oxide of mercury was heated in Expt. 31, the substances produced were different from one another and from the oxide. Red oxide of mercury must evidently contain these different constituents—mercury and oxygen gas—which are not in a merely mixed condition.

Again, when the mixture of iron and sulphur was heated, the product left after heating differed widely from the mixture from which it had been produced.

Hence it is evident that there is a marked difference between substances mechanically mixed and the same substances when they are bound together in chemical combination.

The following experiments will make this difference between a mixture and a compound still more evident.

Expt. 34.—Mix some clean copper filings, which have been sifted through fine muslin, with an equal volume of flour of sulphur. Reserve half this mixture. Heat the remainder in a test-tube, and powder the cool product finely in a mortar. Treat separate portions of the original mixture and of the powdered product from the test-tube as is described below, and note the results which are obtained.

	Before Heating.	After Heating.
a. Note the colour.	The colour is intermediate between red copper and yellow sulphur.	The colour is a deep grey, quite unlike that of the mixture.
b. Examine with a lens.	The separate particles of copper and sulphur are readily seen.	No difference is observed between the separate particles.
c. Heat the substance on a piece of porcelain.	The sulphur burns with a blue flame.	The substance does not burn.
d. Shake up with water, and allow the liquid to stand.	The copper subsides quickly, leaving the sulphur suspended for some time in the water.	No copper or sulphur separates.

From the above experiments it will be seen that the chemical compound of copper and sulphur differs in its properties from its constituents, whereas the mixture retains the properties of the substances which have been mixed to produce it.

Further differences are given in paragraphs 136 and 137.

34. **Simple Substances or Elements.**—Most of the substances which have been used for the preceding experiments can be separated into two or more dissimilar bodies. For instance, when mercuric oxide is heated (Expt. 31), it furnishes the two substances mercury and oxygen. Neither of these two substances, however, has ever been separated into two or more dissimilar substances; they are accordingly called simple substances or elements. Other elements which have already been experimented with are platinum, magnesium, iron, sulphur and copper.

There are some seventy elements known, of which about half are of common occurrence, while the remainder are rarely met with, or are found in small quantity only.

35. Indestructibility of Matter.—In the experiments already described the substances which are used frequently undergo loss or gain in weight. It must be understood, however, that in no case does either destruction of existing matter or creation of fresh matter take place. The apparent destruction or creation of matter is frequently explained by the material undergoing alteration in its condition from a solid to a gas or the converse. The following experiments will further illustrate this indestructibility of matter.

Expt. 35.—Attach an ordinary wax vesta in a vertical position, and with its head upwards, to a block of wood. The match can be made to retain its vertical position by heating the wax for a few moments at the cut end, and then pressing this against the block until the wax has set.

Set fire to the head of the vesta, and while the match is burning, invert over the flame a cold, clean, dry beaker or glass cylinder. Notice that the inside of the jar becomes dimmed by the deposition of water.

Now turn the vessel into the upright position, and pour into it at once some clear lime-water. Shake the lime-water round in the vessel; it will become milky, showing that the air within the jar has been changed by combustion, since ordinary air will not affect lime-water under the above conditions.

Hence it is seen that although the vesta gradually burns away, and appears at first to be destroyed, the matter of which it is composed is in reality only changed into liquid water and an invisible gas which renders lime-water turbid.

If the match is burnt in an enclosed volume of air, and these products are retained and weighed, it may be shown that no alteration is produced in the combined weights of the vesta and of the air by the process of combustion.

Expt. 36.—Cut off about one-third of another wax vesta, selecting that portion which carries the head. Carefully suspend it by a thin copper wire in a thirty-ounce flask, so that the head of the match just touches the bottom of the flask, and secure the upper portion of the wire by pushing it into a cork which fits the neck of the flask air-tight. The length of the wire must be so arranged that when the wire is vertical the head of the match is in contact with the bottom of the flask.

Now weigh the flask and contents, and carefully note down the weight (12–14). Then gently heat the flask where the match touches it, and in this way kindle the match. When the match has ceased to burn, allow the flask to cool, and weigh it once more. The weight will be found to be identical with that previously taken, proving that no loss of weight, and therefore no destruction of matter, has taken place during the process of combustion.

NOTE.—The above experiment is best performed under the supervision of the teacher, since unless the flask is of fair thickness it is liable to burst, owing to the expansion of the air by heat. Even if this should occur, there is no danger, if the flask is held by the neck in such a manner that its body does not face the experimenter.

SECTION IV.

AIR, NITROGEN, AND OXYGEN.

AIR.

36. Action of Air on various Substances.—It has already been shown (**31**) that copper loses its lustre and changes in colour, and magnesium burns brilliantly and is converted into a friable white substance, when it is heated strongly in the air.

Heat alone induces chemical change in many substances; but other substances undergo change only when they are heated in the presence of air. Certain substances undergo change even at ordinary temperature when they are in contact with the air.

If the element sulphur or the element carbon is heated first when air is excluded, and then when air is present, the difference arising from the presence of air will be readily seen.

EXPT. 37.—Select a piece of glass tube about sixteen inches in length and about a quarter of an inch in internal diameter, and make from it two tubes closed at one end, in the way already described in paragraph 24.

Place in the closed end of one of these tubes a piece of sulphur about the size of a pea, and heat the sulphur in the Bunsen-flame (fig. 26, p. 28). The sulphur will melt, and after a time it will pass off as vapour, which will be cooled to a brown liquid on the upper part of the tube. When the sulphur has become cold it will be seen to be unchanged, showing that when the air is practically excluded no appreciable chemical change has occurred to the sulphur.

Expt. 38.—Now place a similar piece of sulphur on platinum foil, or on a slip of porcelain, and hold it over the Bunsen-flame by means of the crucible tongs. The sulphur will melt and kindle, and a sharp pungent smell will be observed to arise from it. Eventually the whole of the sulphur will burn away.

Expt. 39.—Now heat a piece of charcoal in the other glass tube closed at one end, and contrast the result with that obtained by heating a piece of charcoal to redness on platinum foil or on porcelain. No change will occur to the charcoal when air is excluded, but when it is heated with free exposure to air it will slowly burn away, leaving only a trace of white ash.

From the above experiments it is seen that the presence of air is necessary in order to induce chemical change when sulphur and carbon are heated.

37. **A Substance usually undergoes Increase of Weight when the Air acts chemically upon it.**—In the above cases the products of the chemical change are gases, which are not readily weighed. If, however, both the substance and its product are solids, it can be shown that its change is accompanied by increase of weight, showing that the substance takes something from the air.

Expt. 40.—Spread a thin layer of finely-sifted copper or iron filings, or of 'reduced iron,' upon the bottom of a small thin flat porcelain-dish or upon the lid of a porcelain crucible, and weigh the whole (12–14). Then heat the porcelain strongly over the Bunsen-flame, holding it by means of the crucible tongs. Continue the heating for about three minutes, then allow the porcelain to cool, and weigh it again. The weight will now be found to have increased, showing that something has been abstracted from the air by the metal. The substance which has been abstracted will be examined later on. It is known as oxygen.

38. **Action of Phosphorus upon the Air.**—The element phosphorus is a convenient substance for use in studying

the composition of the atmosphere. Care should be taken in handling it, since it readily takes fire and causes painful burns. In order to guard against danger arising from its ready inflammability, phosphorus is usually stored under water.

Expt. 41.—Carefully cut off a piece of phosphorus about the size of half a pea, moistening the fingers and using a wet knife for the purpose. Dry the piece of phosphorus as quickly as possible by pressing it gently between folded filter or blotting-paper.

Place the dried phosphorus upon a white tile or plate, and kindle it by means of a heated wire or glass rod. Immediately place over the burning phosphorus a small bell-jar or a twenty-ounce wide-necked bottle. The phosphorus will burn brilliantly, giving rise to white fumes, which settle like snow on the plate and upon the inside of the jar. Invert the jar and pour water into it; the white substance will readily dissolve.

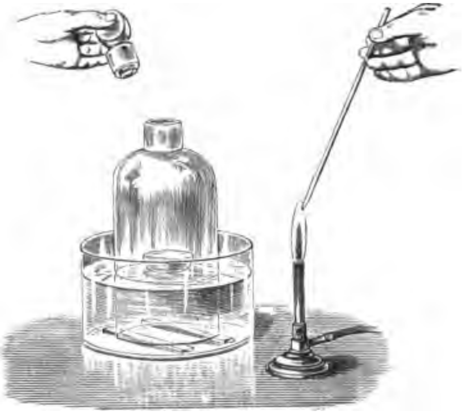
INACTIVE CONSTITUENT OF AIR CALLED NITROGEN.

39. By a modification of the preceding experiment it may be shown that only a portion of the air is removed by burning phosphorus, and that the residual gas is inert in its nature. This experiment may be made by using more phosphorus than can be completely burnt, and kindling it in a vessel the mouth of which is immersed in water. The phosphorus will combine with the whole of the active constituent of the air, and a compound will be formed which will dissolve in the water. A gas will remain with which the phosphorus cannot combine.

Expt. 42.—Float a small dry porcelain-dish, containing a small piece of well-dried phosphorus, upon some water in a pan. Then place an unstoppered bell-jar as a cover over the floating dish, allowing the mouth of the jar to rest upon the bottom of the pan (fig. 28). Kindle the phosphorus by touching it with the end of a long piece of wire, or of a glass rod, which has been heated in the flame. Immediately insert the cork or stopper, and hold down the jar until it stands steadily. When the phosphorus has ceased burning, allow the remaining gas, which has been expanded by heat, to cool; the water will rise in the bell-jar and will fill one-fifth of the space which was previously occupied by the air.

Now pour water into the pan until the level of the liquid inside and outside the bell-jar is the same. Remove the cork or stopper and insert a burning taper; the flame will be at once extinguished, showing the inert nature of the residual gas. This gas is known as nitrogen.

FIG. 28.



PREPARATION OF NITROGEN FROM AIR.

40. Preparation and Properties of Nitrogen.—The gas which remains after the action of phosphorus upon air is not pure nitrogen. It has been shown recently that it contains a small proportion of a still more inert gas called argon, and minute proportions of other gases.

Nitrogen gas may be conveniently prepared by heating a solution of sodium nitrite and ammonium chloride, and may be collected by displacement of water in the apparatus which is described below. The chemical change is as follows :—$NaNO_2 + NH_4Cl = N_2 + NaCl + 2H_2O$.

EXPT. 43.—Fit a four-ounce flask with a doubly perforated cork (27). Pass a thistle-funnel through one hole in the cork, letting it reach nearly to the bottom of the flask. Through the other hole pass a piece of tube which has been previously bent (22) to the shape shown in fig. 29. Now ascertain if the apparatus is sound by closing the mouth of the thistle-funnel by the wetted palm of the hand, and then blowing down the end of the delivery-tube; if no air is heard to escape, the apparatus may be used. If, however, air escapes, the process of fitting must be repeated with another cork.

Now place a small beehive cell in a pan about ten inches in diameter. Pour in water until its surface rises about one inch above the top of the cell, and place the end of the delivery-tube in the inlet of the cell.

Introduce about 5 grams of sodium nitrite and 5 grams of ammonium chloride into the flask, add 50 c.c. water, and gently heat the mixture. When it is thought that the air has been expelled from the apparatus, invert over the beehive cell a glass cylinder, which has been previously filled with water and closed with a glass plate. If this operation is carefully carried out, the jar will remain full of water, and this will be gradually displaced by the gas.

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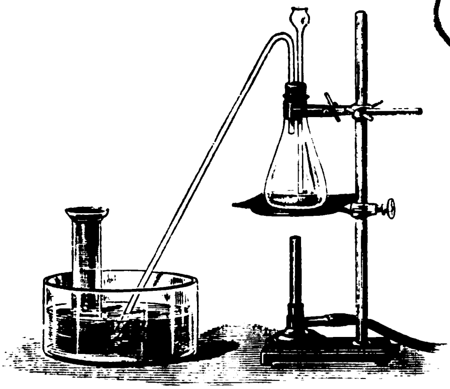
FIG. 29.



PREPARATION OF NITROGEN FROM AMMONIUM NITRITE.

As soon as the jar is full of gas, slide a glass plate over its mouth while it is immersed in the water; remove the jar and place it on one side. Repeat the operation of collection with two fresh jars, and then proceed to examine the properties of the gas as follows, using a fresh jar for each test.

EXPT. 44.—Notice that the gas is colourless, and possesses no smell; that it at once extinguishes a lighted taper; and that when lime-water is shaken in the jar, the liquid remains clear.

THE ACTIVE CONSTITUENT OF THE AIR CALLED OXYGEN.

41. No ready means is known of extracting the nitrogen from air and leaving behind the active constituent, which is known as oxygen. Even the substances which are formed when sulphur, carbon and phosphorus are burnt, do not readily yield up the oxygen which they have taken from the air.

If, however, lead or mercury is heated in the air under suitable conditions it will abstract the oxygen from the air, and the oxygen may be obtained from the product. Mercury may be most conveniently employed for this purpose. The whole of the oxygen which it has absorbed from the air may be obtained from the product, which is commonly known as mercuric oxide.

If the metal is heated strongly in a vessel, which is freely open to the air, its surface becomes slowly coated with red mercury rust, or mercuric oxide. This substance may then be collected and heated more strongly, when it will be decomposed into metallic mercury and oxygen.

42. Preparation of Oxygen from Mercuric Oxide.

EXPT. 45.—Perfectly dry a small test-tube, and place in it sufficient mercuric oxide to cover the bottom. Heat the powder in the Bunsen-flame, *loosely* stopping the end of the tube with the thumb, as is shown in fig. 30. As soon as a grey film, consisting of small drops of mercury, forms upon the inside of the tube, remove the thumb and quickly insert the glowing end of a thin slip of wood into the tube.

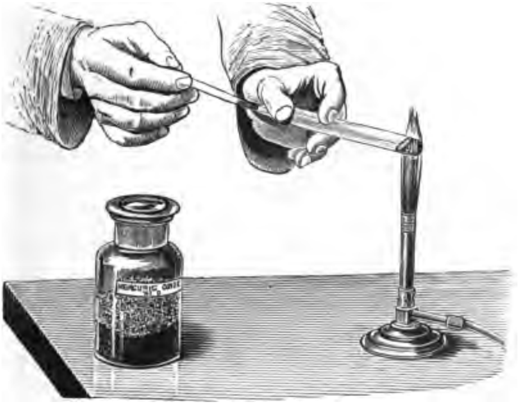
The wood will burst into flame, and will burn more brilliantly than it does in air. This behaviour with a glowing slip of wood is one of the most remarkable properties of oxygen gas, and is frequently used as a *test* for its presence.

The grey film of small globules of mercury, which has collected upon the inside of the tube, may be made to unite into larger globules by rubbing it with a glass rod. The globules of mercury are then easily seen.

The chemical change which has occurred is thus represented by a chemical equation: $2HgO = 2Hg + O_2$.

This method of obtaining oxygen is interesting, because it was the first means known of preparing the gas. It is never used now for preparing oxygen, since less costly substances are known which contain a larger proportion of oxygen than mercuric oxide does, and which give the gas off more easily when they are heated.

FIG. 30.



OXYGEN FROM MERCURY OXIDE.

43. Preparation of Oxygen from Potassium Chlorate.—

Potassium chlorate is usually employed for the preparation of small quantities of oxygen. If it is heated until no more gas is given off, it will have parted with the whole of its oxygen, as is shown by the following equation: $2KClO_3 = 2KCl + 3O_2$.

EXPT. 46.—Heat a few crystals of potassium chlorate in a clean dry test-tube. The white salt will first *fuse* or melt and will then appear to boil. The small bubbles which are given off consist of oxygen gas. This may be readily proved by holding in the mouth of the test-tube a glowing splinter of wood.

44. Preparation of Oxygen from Oxygen Mixture.—Potassium chlorate gives off oxygen gas much more readily than mercuric oxide does. But if the chlorate is mixed with a small quantity of some other suitable substance, which does not itself appear to undergo any permanent change, its oxygen may be driven off by heat with extreme facility. Of these substances manganese dioxide, or black oxide of manganese, is the one usually chosen.

EXPT. 47.—Powder finely in a mortar sufficient potassium chlorate to fill a watch-glass. Mix with it in the mortar about one-fifth as much powdered manganese dioxide, and heat a small quantity of this mixture in a test-tube. The escaping oxygen can be detected by the spark test as soon as the mixture is heated, and a comparatively gentle heat will cause the gas to be rapidly evolved.

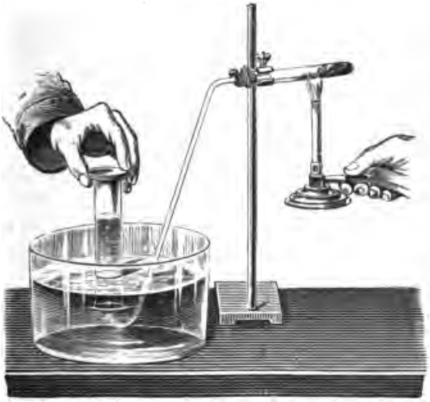
45. Preparation and Properties of Oxygen. — When oxygen gas is required in larger quantity unmixed with air, it is collected over water. For this purpose the oxygen is made to pass through a bent glass delivery-tube, which is fitted by means of a cork into the mouth of the test-tube. The end of this tube dips into some water, and the bubbles of gas as they issue are allowed to rise into a vessel full of water, which is inverted over the end of the delivery-tube. This process of *collecting* oxygen is fully described in the following experiment.

EXPT. 48.—Select a sound cork which is somewhat too large to fit the mouth of the test-tube to be used. Soften it by rolling it on the floor with gentle pressure from the foot. It should now fit tightly into the mouth of the test-tube. Then bend (22) a piece of hard glass tubing, about fourteen inches in length, into the form shown in fig. 31, and make a hole through the centre of the cork (27) of such a size that the glass tube fits tightly into it.

Now fit the apparatus together, and test whether it is air-tight by blowing down the open end of the delivery-tube. Air must not be heard to escape, and it must not be seen to bubble out when the cork is moistened. If air does escape, a fresh cork must be fitted in a similar way.

Now transfer the oxygen mixture into the perfectly dry test-tube, and fit in the cork and delivery-tube. Then proceed to fit up the apparatus as is shown in fig. 31. Fill a pan, about twelve inches in diameter, with water to the depth of about four inches. Support the test-tube either by the hand, or by means of a suitable clamp, so that the end of the delivery-tube dips about one inch below the surface of the water.

FIG. 31.



PREPARATION AND COLLECTION OF OXYGEN.

Next fill the jar, in which the oxygen is to be collected, with water. Close it with a ground-glass plate. Then invert its mouth in the water contained in the stoneware pan, and carefully remove the glass plate. If this operation has been performed with proper care the jar will be entirely filled with water, and will contain no air-bubble.

Now remove the delivery-tube from the water, and heat the front part only of the oxygen mixture, since the escape of gas from the hinder portion would force the mixture out of the tube. A small flame should be used, and it should be slowly moved from side to side in order to lessen the risk of cracking the glass.

As soon as a glowing slip of wood is kindled when it is held at the mouth of the delivery-tube, the oxygen has driven out the air and is beginning to escape. The end of the delivery-tube is then at once dipped under water beneath the mouth of the jar, and the ascending bubbles will rapidly displace the water. A stoneware beehive-shelf will be found convenient for supporting the jar: this is shown in fig. 29. As soon as the vessel is full of gas, close its mouth under water with the glass plate, and remove it for experiment.

Four jars filled with oxygen will be required.

If at any time the gas should be given off too rapidly, the flame must be removed until the current of gas slackens. When the process is to be stopped, the end of the delivery-tube must be removed from the water before the gas has ceased to bubble out, else the water might recede into the hot test-tube.

Oxygen gas is remarkable for the energy with which it combines with or burns many substances. Three examples of this property are given in the following experiments.

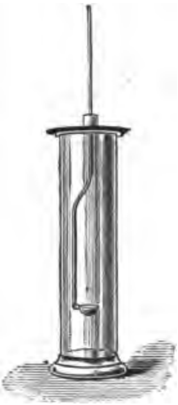
Expt. 49.—Place a piece of **Wood-charcoal** about the size of a nut in a metal deflagrating spoon. Then adjust the wire handle in the brass cap, so that when the spoon is placed in the jar it is about an inch from the bottom (fig. 32). Heat the charcoal in the Bunsen-flame until a part of its surface glows, and quickly place it in the jar of oxygen. The charcoal will burn much more brilliantly and rapidly than in air, and will combine with the oxygen and convert it into carbon dioxide gas: $C + O_2 = CO_2$. As soon as the combustion ceases, pour into the jar some clear lime-water from a small beaker. Then quickly close the jar and shake the liquid inside it. The clear liquid will become milky.

This indicates the presence of carbon dioxide gas, as will be hereafter explained.

Expt. 50.—Remove the charcoal from the deflagrating spoon, and replace it by a piece of **Sulphur** as large as a pea. Heat the spoon in the flame until the sulphur melts and burns with a pale blue flame. Then place the spoon in a fresh jar of oxygen. The sulphur will at once burn with a much brighter flame, converting the oxygen into sulphur dioxide gas: $S + O_2 = SO_2$. The presence of this gas is proved by its suffocating smell.

Pour a little water into the jar and shake it round. Sulphurous acid will be formed and may be recognised by dropping into the water a piece of blue litmus paper, which is immediately reddened; also by pouring in *a few drops* of red potassium dichromate solution, the colour of which changes to green.

Fig. 32.



Combustion in Oxygen.

Expt. 51.—Burn away any sulphur which remains in the deflagrating spoon and place in it *when cold* a small piece of *carefully dried* **Phosphorus** not larger than half a pea. The precautions stated in Expt. 41 (38) must be observed in dealing with the phosphorus. Set fire to the phosphorus by holding the spoon in the flame and notice how it burns in the air. Then place the spoon in a jar of oxygen.

The phosphorus will burn much more brilliantly, producing white phosphoric oxide: $P_4 + 5O_2 = P_4O_{10}$. As soon as the phosphorus has ceased burning, pour in a little water and shake it round in the jar. The white substance will dissolve, yielding phosphoric acid: $P_4O_{10} + 6H_2O = 4H_3PO_4$. The water may now be proved to contain an acid by dropping into it a piece of blue litmus paper, which will be immediately reddened.

Tests for Oxygen.—Oxygen is conveniently recognised by introducing into the gas a slip of wood with a spark at the end; the wood will burst into flame. This test does not answer if the oxygen is largely mixed with another gas.

Nitrogen monoxide (114) also causes a spark to burst into flame, but the flame is surrounded by a halo in this gas.

A more delicate test for oxygen depends upon its power of producing a reddish-brown gas when it is mixed with nitric oxide (116, 117).

QUANTITATIVE EXPERIMENTS WITH POTASSIUM CHLORATE.

46. **The Weight of Oxygen which is contained in Potassium Chlorate** may now be determined. This is effected by weighing some potassium chlorate, and then heating it until the whole of the oxygen which it contains has been evolved as gas. The residual salt is then weighed. The loss of weight which the original chlorate has suffered by being heated, will be the weight of oxygen which it contained.

EXPT. 52.—Close one end of a piece of combustion tube about five inches long, by drawing it out in the *foot blow-pipe-flame* (24). Weigh the tube as soon as it is cold, and introduce into it one gram of dry potassium chlorate, taking care that the whole of the salt passes down to the closed end of the tube. Now hold the tube in an inclined position and heat the chlorate in the Bunsen-flame until oxygen gas ceases to come off. Then allow the tube to cool, and weigh it again with its contents. In order to make sure that the whole of the oxygen has been expelled from the chlorate, heat the salt again, allow it to cool, and once more weigh it. Repeat these operations until two successive weighings give identical results. The loss of weight which the tube and its contents have suffered will give the weight of oxygen which was present in one gram of the chlorate.

The weighings should be entered in the note-book in the following form :—

		Grams.
Weight of tube + potassium chlorate	=	23·53
Weight of tube alone	=	22·53
Weight of potassium chlorate used	=	1·00

Grams.
Weight of tube + contents after first weighing = 22·20
,, ,, second ,, = 22·16
,, ,, third ,, = 22·16

Gram.
Weight of Oxygen driven off = 23·53 − 22·16 = 0·37

Gram.
Weight of Oxygen calculated from the formula $KClO_3$ = 0·39
Weight of Oxygen found . . . . . = 0·37

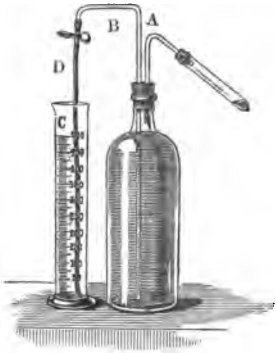
Experimental error . . = −0·02

47. Measurement of the Volume of Oxygen evolved by heating Potassium Chlorate.

47. Measurement of the Volume of Oxygen evolved by heating Potassium Chlorate.—The volume of gas which is evolved from a substance is most easily measured by causing the gas to displace its own volume of water, and then measuring the water which has been displaced.

The apparatus described below will be found convenient for this purpose. In order to secure comparable volumes of gas in this process by correction, care must be taken that the gas is under the same pressure as the atmosphere before the displaced water is measured, and that the temperature of the gas is noted when the volume is read (see Note, p. 50).

Fig. 33.



Measurement of Oxygen from Potassium Chlorate.

Expt. 53.—Fit into the neck of a Winchester-quart bottle a rubber cork which has been bored with two holes. Through one hole insert a short piece of glass tube bent at an angle (fig. 33, A). This tube should pass only just through the cork. Through the other hole pass a glass tube (B) bent twice at right angles, the longer end of which reaches to the bottom of the bottle. To the shorter end of this tube attach a piece of rubber tubing (D), which can be closed by means of a pinchcock.

Now powder some potassium chlorate ($KClO_3$), and dry it, if necessary, for half an hour in the steam-oven (189, fig. 64). Weigh out one gram of the dry powder into a piece of combustion-tube closed at one end (Expt. 52, 46), which has been fitted with a cork suitably bored for the insertion of the free end of the small piece of glass tube (A). Then pass the end of the clamped rubber tube (D) to the bottom of the graduated cylinder (C).

A plain cylinder may be used instead of the graduated one, since the water which it receives can be measured, at the end of the experiment, in another graduated vessel.

Ascertain that all the connections are gas-tight, and then proceed to make the determination as follows. Fill the bottle with water, detach the tube which contains the potassium chlorate from the cork. Insert the cork and tubes into the bottle and gently blow through the short tube (A) until the water commences to flow from the lower end of the rubber tube, then clamp the upper end of this tube. The tubes (BD) will now be full of water. Place the end of the rubber tube (D) in the cylinder, attach the combustion-tube to (A), and open the clamp.

Now gently heat the tube so as to fuse the salt, and gradually increase the temperature until oxygen escapes from the salt with effervescence. As soon as the effervescence has ceased, and no more water is displaced from the bottle into the cylinder, allow the heated tube to cool. Then lower or raise the cylinder (*c*) until the level of the water is the same in the cylinder as in the bottle. At once close the clamp and read the volume of water in the cylinder. This will be the same as the volume of the oxygen which has displaced it.

NOTE.—The result which is obtained in the above experiment will be influenced by the effect of temperature on the volume of the gas. An increase of temperature causes an increase in the volume of the gas, and a decrease in the temperature a decrease in the volume. In order to make the results of different experiments comparable one with another it is usual to apply a correction which reduces the volume of the gas to that which it would occupy at 0° C. The correction for differences in barometric pressure and in the pressure of aqueous vapour need not be taken into consideration, since the errors of the experiment will probably affect the gaseous volume to a greater extent than these corrections would do.

The following is the method of calculating the volume which the gas would have at 0° C.:—

Let t be the temperature of the oxygen in the bottle at the end of the experiment, and V the volume of the water displaced:

Then the volume of the oxygen at 0° C. would be $\frac{273V}{273+t}$.

FURTHER EXPERIMENTS WITH AIR.

48. Composition of Air by Volume.—The proportion of nitrogen to oxygen in air was roughly determined in Experiment 42 (39), and was found to be four volumes of nitrogen to one volume of oxygen. Accurate analyses show that the proportion by volume is 79·1 per cent. nitrogen and 20·9 per cent. oxygen.

Although the method described below is not sufficiently accurate to give the percentage with absolute correctness, still it will be found to be more accurate than the method of burning phosphorus in the air.

EXPT. 54.—Select a piece of glass tubing about twenty-four inches in length and half an inch in internal diameter. Close one end in the blowpipe-flame (24), and carefully fit into the open end a rubber cork which enters about one-quarter of its length.

Weigh out about a gram of pyrogallic acid, and dissolve it in 10 c.c., or about half a test-tube full, of water. Pour this solution into the long tube, quickly drop in a piece of stick potash half an inch in length, and immediately insert the cork. Invert the tube and mark the surface of the solution with a rubber band. Now cause the liquid to flow gently from end to end of the tube for about five minutes. By this time the oxygen will have been absorbed by the alkaline solution of potassium pyrogallate, and the nitrogen only will remain.

Now immerse the end of the tube in a cylinder of water and remove the cork under the water. Lower the tube into the water until the level of the liquid is the same on the outside as in the inside of the tube, and slip on a tight rubber band to mark the altered level of the liquid in the tube. The ratio of the volume of oxygen to that of nitrogen will be as the distance between the lower and upper band is to the distance from the upper band to the top of the tube.

Calculate from this proportion the percentage of oxygen in the air: that is, the number of volumes of oxygen in 100 volumes of air.

OTHER CONSTITUENTS OF AIR.

49. Air contains small quantities of other substances in addition to the main constituents nitrogen and oxygen. The most important of these are water vapour and carbon dioxide gas, but other constituents have already been referred to (40).

50. **Moisture** may be shown to be present in air by condensing the water vapour into the liquid state by means of cold.

EXPT. 55.—Carefully dry the exterior of a beaker, and place in it a few crystals of sodium thiosulphate. Pour in some water and stir it with the crystals, so as to promote the solution of the substance. The temperature of the water will be very considerably reduced by the process of solution, and after a few minutes the *exterior* of the beaker will become covered with moisture. This is due to the cold surface of the glass condensing the water vapour of the air with which it is in contact.

51. **Carbon dioxide** is present in fresh air in very small proportions, but the amount increases in closed unventilated

rooms in which persons are breathing and flames are burning. The presence of this gas in air may be shown by its power of producing turbidity in lime-water with which it is brought into contact (80, 81).

Expt. 56.—Pour a little clear lime-water into a watch-glass, or clock-glass, and expose it to the air for about half an hour. At the end of that time a film will be seen upon the surface of the lime-water. This is due to the action of carbon dioxide upon the lime. The film may be broken up into white flakes by stirring the liquid.

SECTION V.

COMPOSITION OF WATER.
HYDROGEN.
NATURAL WATERS.
SOLUTION, DISTILLATION.
EVAPORATION AND CRYSTALLISATION.
PRECIPITATION, FILTRATION, AND DECANTATION.

WATER.

52. **Water** differs chemically from air in being a compound of two gases, oxygen and hydrogen, whereas air was shown to be a mixture of the gases nitrogen and oxygen.

No simple method is known of forming water from its constituents and collecting the product. But it may be easily shown that hydrogen is a constituent of water, by causing a suitable metal to combine with the oxygen present in the water and thus to liberate the hydrogen.

53. **Hydrogen liberated from Water.**—One of the most convenient metals for liberating hydrogen from water is the metal sodium. When this metal is thrown upon water it energetically unites with the oxygen, and liberates the hydrogen. The action may be moderated by dissolving the sodium in mercury, so as to produce some 'sodium amalgam' in the liquid metal.

EXPT. 57.—Place a small quantity of mercury in a mortar, and drop upon it, from time to time, a thin freshly-cut slice of sodium. Press down each slice of metal firmly with the pestle until combination takes place with a hissing noise and a glow. Carefully avoid inhaling the poisonous mercury vapour which is given off. When a suitable quantity of sodium has been introduced, the cold solution of the amalgam will be slightly pasty in consistency. Now partially fill a small porcelain-dish with water, and pour in a small quantity of the sodium amalgam. Bubbles of gas will be given off from the surface of the amalgam.

Note.—The small dishes which are used by the metallurgist, and are known as roasting-dishes, answer very well for this purpose. They are flat-bottomed, and stand more steadily than a porcelain-dish does.

Proceed to collect the gas which is evolved as follows. Fill a test-tube with water, close the mouth of the tube with the thumb, and invert the tube in the water, with its mouth over the amalgam. As soon as the tube is about half full of gas, close the tube by placing the thumb over its mouth, and remove it from the water. Now invert the tube, remove the thumb, and immediately apply a light. The gas will be kindled, and will burn with the pale flame which is characteristic of hydrogen.

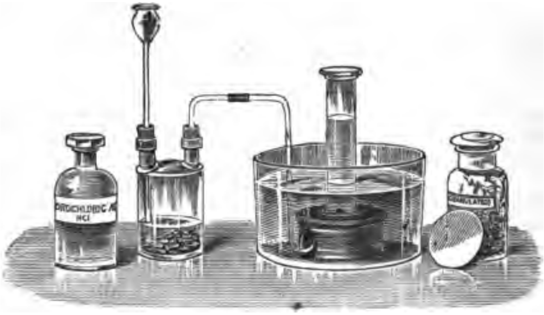
When hydrogen is burnt in air, the substance formed is water (55). Since oxygen has been shown to be the active constituent which maintains combustion in the atmosphere (41), it is evident that water is composed of the gases oxygen and hydrogen.

Collection and Properties of Hydrogen.

54. **Hydrogen** is most readily prepared by the interaction between a suitable acid and a suitable metal. For most purposes the acid used is hydrochloric acid, and it is allowed to act upon zinc.

Expt. 58.—Fit a two-necked Woulffe's bottle with perforated corks, one of which bears a thistle-funnel whose end reaches nearly to the bottom of the bottle, and the other a bent tube terminating just below the cork (fig. 34). Then adapt a bent delivery-tube by means of a piece of rubber tubing. A wide-necked bottle, fitted as is shown in fig. 48 (p. 80), may be used instead of the Woulffe's bottle.

FIG. 34.



PREPARATION OF HYDROGEN.

Place sufficient granulated zinc in the bottle to cover the bottom, replace the cork, and ascertain that the apparatus is air-tight by closing the end of the delivery-tube and blowing down the thistle-funnel. Then pour water in through the funnel until the zinc and the end of the funnel-tube are covered, and add pure strong hydrochloric acid gradually (*Caution*, p. 26). Hydrogen will be seen rising from the zinc in numerous small bubbles: $Zn + 2HCl = H_2 + ZnCl_2$.

Since the acid is decomposed during this reaction, it is necessary to pour in more acid occasionally through the funnel in order to maintain the evolution of the gas.

Dip the end of the delivery-tube under water contained in a pan, and allow the gas to bubble out through the water for at least five minutes. This delay in collecting the gas is necessary in order to give the hydrogen time to remove the air which filled the bottle.

Since a mixture of air with hydrogen burns explosively when it is kindled, it is well to make sure that the air has been completely removed from the bottle, by making the following test on a small quantity of the escaping gas. Invert a test-tube filled with water over the end of the delivery-tube. Close the test-tube with the thumb when it is full of gas; remove it from the water, and hold its open mouth to a flame. If the gas burns with a slight explosion, the tube is again filled with the gas and the experiment is repeated. As soon as the gas burns quietly with a pale flame, a small thick-glass cylinder may be filled with hydrogen in the same way as was directed for oxygen (45).

Expt. 59.—Hold the cylinder, which contains the hydrogen, mouth downwards; remove the plate, and at once push up a lighted taper into the gas. The gas will burn with a pale flame, and will extinguish the flame of the taper.

Expt. 60.—Hold another jar of hydrogen for a short time with its mouth open and directed upwards. The gas will entirely escape, as may be proved by holding a lighted taper in the vessel, when no hydrogen flame will be seen.

If the cylinder is once more filled with hydrogen and is then held for a short time with its mouth downwards, the hydrogen will remain in it and will burn with a pale flame when a lighted taper is introduced. Note, however, that the gas burns in this case with a slight report; this is due to air having mingled with the hydrogen by spontaneous *diffusion.*

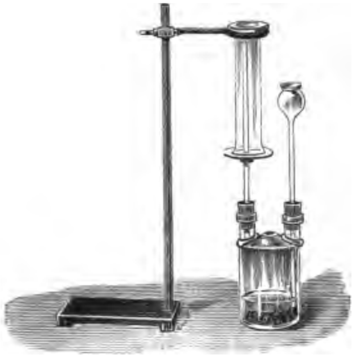
Since hydrogen does not quickly escape downwards, but rapidly escapes upwards, it must be lighter than air.

Advantage is often taken of the lightness of hydrogen to make it replace air instead of water in the vessel in which it is to be collected. For this purpose a straight delivery-tube from the hydrogen-apparatus is passed up to the top of an inverted jar; the hydrogen then gradually pushes down the heavier air and fills the jar by displacement.

Expt. 61.—Fill a jar with hydrogen by displacement in the following way: quickly replace the bent tube in the cork of the Woulffe's bottle by a straight one, eight or nine inches in length (fig. 35). As soon as a brisk effervescence of gas has been caused by pouring in some strong hydrochloric acid, pass this tube up to the top of an inverted jar; and allow the jar to remain in this position for several minutes.

Now remove the jar, keeping it inverted, and gradually bring it into an erect position while its mouth is near a flame. The light hydrogen will rise out of the jar and will burn in the air.

FIG. 35.

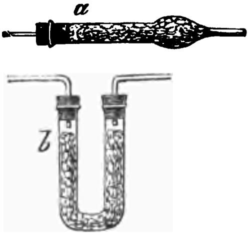


COLLECTION BY DISPLACEMENT.

55. Hydrogen forms Steam by its Combustion.—This may be shown by burning the hydrogen and condensing the steam into water by contact with a cold surface.

EXPT. 62.—Cover the Woulffe's bottle and the funnel-tube with a cloth, in order to prevent accident in case of an explosion, and light the hydrogen at the end of the delivery-tube used. Hold over the flame a tumbler or beaker, which is perfectly clean, dry, and cool. The inside of the vessel will become dimmed with moisture, showing that when hydrogen gas is burnt in air it unites with the oxygen and produces water vapour: $2H_2 + O_2 = 2H_2O$.

FIG. 36.



DRYING TUBES.

In order to make the above results conclusive the hydrogen should be dried before it is burnt, and the moisture should be proved to be pure water.

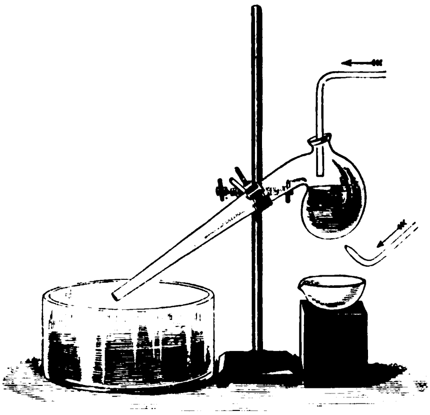
The gas may be dried or *desiccated* by passing it through some substance which readily absorbs moisture, such as strong sulphuric acid or calcium chloride. A straight tube containing fragments of calcium chloride (fig. 36 *a*), or a U-tube filled with pieces of pumice-stone moistened with strong sulphuric acid (*b*), may be connected with the hydrogen apparatus for the purpose of drying the gas.

The amount of water which is deposited upon the cooled beaker is too small to allow of its purity being ascertained, but a larger quantity may be obtained by allowing the hydrogen flame to impinge for some time upon a cold surface, as is directed below.

EXPT. 63.—A convenient way of collecting water from the combustion of hydrogen is shown in fig. 37. An eight-ounce retort is supplied with a constant stream of tap-water, the water entering through the tubulure by means of a rubber tube, and overflowing to the sink through the neck. The hydrogen flame is made to play against the bulb of the retort, and the condensed water which drops from the bottom of the retort is collected in a clean dish.

The liquid which is thus obtained may be shown to be water by possessing neither taste nor smell, and by the fact that dried copper sulphate is immediately turned blue when it is moistened with the liquid. This latter result is given by water only (Expt. 78, p. 69).

FIG. 37.



SYNTHESIS OF WATER.

EXPT. 64.—Fill a short thick-glass cylinder with water, and invert it in a pan of water. Displace two-thirds of the water in the cylinder with hydrogen (Expt. 58, p. 55), and the remainder with oxygen (Expt. 48, p. 45). Let the jar stand with its mouth under water for five or six minutes in order to allow the gases to mix. Then raise the

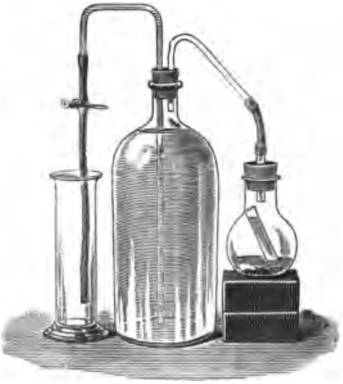
inverted jar and apply a lighted taper: the gases will combine to form water with a loud explosion.

56. Tests for Hydrogen.—Hydrogen gas is recognised by burning with a pale flame in air or in oxygen, the flame of the dried gas depositing water on any cold object which is held above it. It also explodes with formation of moisture, when it is mixed with oxygen or with air and fired.

57. Estimation of the Volume of Hydrogen which is evolved by the action of Hydrochloric Acid upon Zinc.—A similar apparatus to that which has been described in Expt. 53 (p. 49) may be used for this estimation, but the test-tube is replaced by a flask.

Expt. 65.—Weigh out accurately one gram of zinc and place it in a six-ounce wide-necked flask, which has been fitted with a perforated rubber cork, carrying a short upright tube. Add about 14 c.c. of water, and introduce carefully into the flask a test-tube three inches long by half an inch broad, which has been almost filled with strong hydrochloric acid (*Caution*, p. 26).

Fig. 38.



Volume of Hydrogen separated from Acid by Zinc.

Connect the flask with the tube of the aspirator by means of a rubber joint. The apparatus will now be arranged as is shown in fig. 38.

Ascertain that the fittings are air-tight, and then tilt the flask so that the acid runs out of the test-tube into the flask, and comes into contact with the zinc. When no more gas is evolved, collect the water which has been displaced by the gas in a glass cylinder, measure it, and correct the volume for temperature, as has been described in the note, paragraph 47.

NATURAL WATERS.

58. Water is never found in nature perfectly free from dissolved solids. Even carefully collected rain-water contains some substance in solution. Spring-water, river-water, and well-water usually contain some solid matter, which they have dissolved from the soil or rock with which they have been in contact.

The amount of solid matter which is dissolved in water can be readily determined by boiling a measured volume of the water to dryness and weighing the residue.

EXPT. 66.—Weigh a small porcelain-dish and measure into it 50 c.c. of tap-water (9). Then heat the dish by means of a small Bunsen-flame, as is described in Expt. 33 (page 32), carefully avoiding vigorous ebullition, which would cause the liquid to spirt out of the dish and would lead to loss of dissolved matter. If the dish is heated over the water-bath (66), all risk of such loss will be avoided. As soon as the contents of the dish are dry, weigh the clean dry dish again. The increase of weight will be the weight of the solid matter which was present in the 50 c.c. of tap-water.

In order to free water from dissolved solids, it is subjected to 'distillation.'

DISTILLATION.

59. The process of Distillation serves to separate liquids, which boil at a comparatively low temperature, either from solids, or from other liquids which are only converted into vapour at a much higher temperature. It consists in boiling

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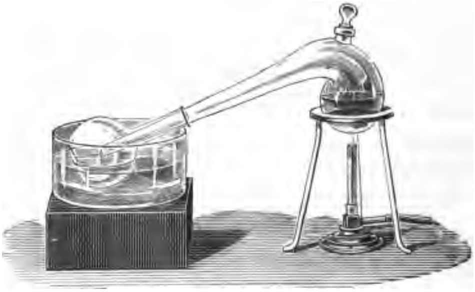
the liquid, and cooling or *condensing* its vapour into a liquid which is termed *the distillate.* The non-volatile solid or liquid substances are left behind in the vessel in which the liquid is boiled. The purification of common spring-water from the solid substances which are dissolved in it, will serve as an example of this process.

EXPT. 67.—**Distillation of Water.**—Pour tap-water into a clean eight-ounce retort (fig. 39) through a funnel placed in the tubulure, until the retort is half full. Support the retort on the ring of a retort-stand or upon a tripod-stand, with its neck sloping downwards, and push the neck into a small clean flask, which is partly immersed in cold water contained in a pan or bowl. Now carefully boil the water in the retort, avoiding such tumultuous agitation of the liquid as would project it into the neck of the retort. Steam will pass into the flask and will be there condensed to *distilled water.* Shake the first portion of the distillate round in the flask and throw it away, as it is apt to have been rendered impure by rinsing the neck of the retort and the flask.

FIG. 39.



DISTILLATION OF WATER.

60. Tests for Salts dissolved in Tap-water.—The following tests serve to indicate the presence of some of the salts which occur most frequently in ordinary water-supplies.

Any or all of these salts may be almost or entirely absent from any particular water-supply.

EXPT. 68.—While the process of distillation is going on, separate portions of the tap-water may be subjected in separate test-tubes to the following tests :—

Chloride.—Add several drops of nitric acid and of silver nitrate solution. If the water becomes milky, the presence of chloride in the water is indicated.

Calcium Salts.—To a separate portion of tap-water in another test-tube add some ammonium hydrate and some ammonium oxalate solution. The presence of calcium salts is indicated by the production of turbidity.

Sulphate.—The presence of sulphate is proved if turbidity is caused by adding a few drops of hydrochloric acid and of barium chloride solution to another portion of the tap-water.

If separate portions of the distilled water from the flask are examined by the same tests they will remain clear, since any of the above solid substances, which were present in the water, will have been removed by distillation.

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Hardness.—The presence of calcium or magnesium salts in the tap-water causes the precipitation of soap. Water which produces this result is termed 'hard.' Sodium chloride and certain other salts, if present in large amount, may also cause the precipitation of soap.

Dissolve a small shaving of ordinary soap by warming it in a little distilled water. Add a few drops of this soap-solution to some tap-water which half fills a test-tube; close the mouth of the test-tube with the thumb, and shake the water well. If no lather forms, and the water becomes turbid owing to the separation of the soap, the tap-water is 'hard.' In this case, repeat this experiment, substituting an equal quantity of distilled water for the tap-water, and adding soap-solution to it in the same quantity as before; a lather will be formed when the water is shaken, and the liquid will not become turbid.

61. Tests for Impurities in Distilled Water.—Distilled water should leave no residue when it is evaporated to dryness. When solutions of ammonium oxalate, silver nitrate, barium chloride, and ammonium sulphide are separately added to different portions of the water, no turbidity or darkening of colour should be produced. These tests prove the absence of calcium salts, of chlorides, of sulphates, and of lead and iron salts.

SOLUTION.

62. Solution.—Many solid substances, when they are stirred or shaken with water or with some other liquid, gradually *dissolve*. The liquid which is thus obtained is called a *solution* of the solid, and the liquid which dissolves the solid is termed the *solvent*. Further, a solid which dissolves in a liquid is said to be *soluble* in that liquid; if it does not dissolve, it is said to be *insoluble*.

The process of solution is most rapid when the solid substance is *in the state of powder*. The process is also much hastened by heating the solvent.

Water is the solvent which is most frequently used, but some bodies are insoluble in water, and others dissolve only slightly. Most substances are more easily and largely soluble in hot than in cold solvents (67).

Some bodies, which are insoluble in water, are readily soluble in other liquids.

63. Simple Solution occurs when a substance dissolves in a liquid without undergoing alteration in composition. The solid substance is therefore obtained again unchanged in composition when the solvent is removed by the *evaporation* of the solution.

EXPT. 69.—Powder a little copper sulphate in a mortar, and transfer it to a small porcelain-dish. Half fill the dish with water, and heat it with a small flame upon a pipe-clay triangle placed on a tripod or retort-stand. The *blue* copper sulphate will dissolve, yielding a *blue* solution. Keep this solution for Expt. 74.

64. **Chemical Solution,** on the other hand, is always attended by a chemical change in the substance which has been dissolved. The solution therefore contains a substance differing in composition from the undissolved solid, and when the solvent is removed by evaporation (66) the original substance is not obtained.

EXPT. 70.—Place a small piece of marble (calcium carbonate) in a test-tube, pour in a little water, and heat the tube. The calcium carbonate will not disappear. Pour off this liquid and keep it.

Now add some hydrochloric acid: *effervescence*, or escape of numerous small bubbles of gas, will occur. This gas will render lime-water turbid, and is therefore carbon dioxide gas. The piece of calcium carbonate will meanwhile slowly diminish in size, and will at last entirely disappear in the liquid, if sufficient acid has been added. Keep this solution.

This is an example of chemical solution. The calcium carbonate is changed by the hydrochloric acid into calcium chloride, and this substance, and *not the original calcium carbonate*, remains in solution.

It will be noticed that a gas is given off. This is a very usual, but not a universal, effect of chemical solution.

65. **Action of Water and of other Liquids upon Substances.**—The effect of treating various substances with water and with acids may now be studied.

Heat a small quantity of a powdered solid with water, and note whether the substance dissolves entirely.

If the substance has not dissolved, a few drops of the clear liquid may be evaporated to dryness by gentle heat in a watch-glass; if a residue is left, a portion of the substance must have been dissolved.

This is confirmed if the water has become coloured by contact with the substance, or if blue litmus or turmeric test-paper (145) becomes changed in colour when it is dipped into the liquid.

Treat separate portions of the substance in a similar manner with each of the common dilute acids and subsequently with the same acids in an undiluted condition. Notice whether the substance is partially or wholly dissolved; also whether any gas is given off; note also any other changes which occur.

For practice in the method observe the effect (1) of water, (2) of dilute hydrochloric acid, and (3) of dilute sulphuric acid on calcium carbonate.

Expt. 71.—Pour a few drops of the water, which has been heated with calcium carbonate (Expt. 70), into the hollow of a watch-glass and heat the glass at some distance from a small flame until it is dry; no solid residue will remain on the glass, showing that calcium carbonate is insoluble in water.

Evaporate a few drops of the solution of marble in hydrochloric acid (Expt. 64) in a similar way; white calcium chloride will remain. Marble has been shown to be completely soluble in hydrochloric acid.

Pour some dilute sulphuric acid upon a small quantity of powdered marble in a test-tube and heat the liquid. The powder will effervesce slowly, and will give off carbon dioxide gas, but the powder will not disappear even when more acid is added. Allow the liquid to stand until it is clear, and then evaporate a few drops upon a watch-glass. A white residue will remain. In this case a white powder remains undissolved by the acid, and some of the same substance passes into solution and is obtained by evaporating the liquid. This is due to the conversion of the calcium carbonate into calcium sulphate, which then passes partially into solution.

Expt. 72.—Try the effect of water, dilute hydrochloric acid, dilute sulphuric acid, and dilute nitric acid respectively upon a small portion of each of the following substances, and note down the results obtained: calcium oxide, copper oxide, lead oxide, and zinc.

Saturated Solution.—The amount of a solid substance which can undergo simple solution in any particular solvent usually increases with the increase of temperature of the solvent. But there is always a limit to the amount of the solid which can be dissolved by a particular volume of the solvent at any one temperature. A solution which contains this maximum possible amount of the solid is known as a 'saturated solution.' A method for estimating this amount is given in the next paragraph.

EVAPORATION.

66. Evaporation.—When it is wished to obtain a substance, which is dissolved in a liquid, in the solid condition, the liquid is removed as vapour, or *evaporated.* The solid substance is then left behind in the vessel.

A solution is usually evaporated by heating it in a porcelain-dish. A Bunsen-flame is commonly employed as the source of heat. The flame should be reduced in size towards the end of the process, in order to prevent the liquid and the substance from spirting out of the dish. The dish may also be heated by means of steam by placing it upon a water-bath (fig. 40, p. 68); the process is thus rendered much less rapid, but risk of spirting is avoided.

As an exercise in evaporation, the amount of a solid substance which water can dissolve at any particular temperature may be ascertained. For this purpose a measured quantity of the saturated solution is evaporated to dryness and the residue is weighed.

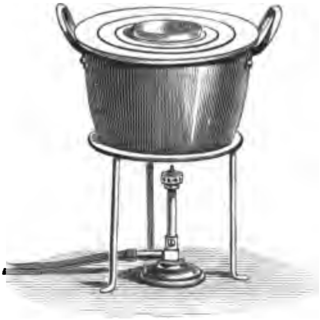
Expt. 73.—Half fill an eight-ounce beaker with distilled water and heat the water nearly to boiling. Then drop in powdered potassium chlorate in small successive quantities until the last portion is not dissolved. Now allow the liquid to cool to the temperature of the laboratory, which should be noted. A portion of the dissolved salt will separate from the solution, because potassium chlorate is more soluble in hot than in cold water.

Remove 25 c.c. of the clear solution, by means of a pipette, to a weighed porcelain-dish, and weigh the dish with the liquid which it contains. Evaporate it carefully to dryness. Allow the dish to cool, and then weigh it. The increase of weight of the dish will correspond to the weight of the salt which was dissolved in 25 c.c. of the liquid. From this result the percentage weight of salt, which was dissolved in the saturated solution at the particular temperature noted, may be calculated, since the weight of water which dissolved the known weight of the salt will be equal to the difference between the weight of the dish with solution and the weight of the dish with the salt.

CRYSTALLISATION.

67. **Crystallisation.**—A part of the liquid may be evaporated from a solution for the purpose of *concentrating* it. When the hot solution has been sufficiently concentrated, it will frequently deposit *crystals* of the dissolved substance while it is cooling.

FIG. 40.



THE WATER-BATH.

If the process of crystallisation is allowed to take place slowly, the crystals will be large and distinct, and will be seen to possess a definite geometrical shape.

EXPT. 74.—Concentrate the copper sulphate solution (Expt. 69, p. 64), and allow the liquid to cool. If sufficient water has been evaporated, crystals of copper sulphate will form. Keep the solution for Expt. 81.

Expt. 75.—Half fill an eight-ounce beaker with water, and heat the liquid to boiling. Add small successive quantities of powdered alum, until the last portion which is added is not dissolved. Then allow the solution to cool. Colourless and transparent crystals will be deposited, and each of them will be in the form of a more or less perfect regular octahedron.

If the crystals are small, a lens may be used to observe their shape and appearance.

Expt. 76.—Crystallise from water the following salts and note the shape and colour of the crystals.

Potassium chlorate. Magnesium sulphate. Lead chloride. Potassium dichromate. Potassium ferrocyanide.

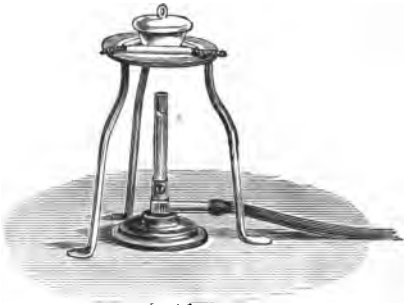
68. Water of Crystallisation.—Many substances retain a definite proportion of water when they are crystallised from their solution in water. This water appears to be chemically combined with the substance. It is known as water of crystallisation. It is, as a rule, readily driven off by heat. If the crystallised salt is coloured, it frequently loses its colour when the water of crystallisation has been driven off, and the dry salt regains its colour again when it is moistened.

Expt. 77.—Heat a crystal of washing soda, or sodium carbonate, in a test-tube held in a horizontal position. The crystal will quickly melt; the liquid will then boil and water will be seen to condense in the upper part of the tube. The amount of water present in clear washing-soda crystals amounts to more than half the weight of the crystals. This may be proved by treating the crystals as is described in Expt. 79 for barium chloride crystals.

Expt. 78.—Heat a crystal of copper sulphate; water will be given off from the blue crystals, and the residue will be white. When the residue is cold, moisten it with water; the salt will regain its blue colour.

Expt. 79.—Weigh a porcelain crucible and gradually introduce powdered crystallised barium chloride into it until its weight is increased by exactly two grams. Support the uncovered crucible upon a pipeclay triangle on a tripod-stand (fig. 41), and heat it for about fifteen minutes. Then cover it, allow it to cool, and weigh it again. The whole of the water will have been driven off from the crystals, and the loss of weight suffered by the crucible and salt will give the weight of water contained in two grams of the crystallised salt. From these numbers the percentage weight of the water of crystallisation in the crystals may be calculated.

FIG. 41.



HEATING IN CRUCIBLE.

PRECIPITATION.

69. Precipitation.—It has already been stated that many substances are insoluble in water. By mixing two perfectly clear and transparent solutions together it is possible to produce a substance which is insoluble in the liquids which are present. When this occurs the liquid becomes more or less turbid or opaque, owing to the separation of a solid substance. A solid substance which has been thus produced in a liquid is called a *precipitate*.

EXPT. 80.—Pour into a test-tube a little barium chloride solution, and add to it some solution of ammonium carbonate. A white precipitate of barium carbonate will be produced. Keep this test-tube with the precipitate.

A substance is often purposely removed from its solution by causing it to form an insoluble compound or precipitate. The substances which are added for this purpose are termed *reagents*; they are commonly liquids.

In producing a precipitate, care must be taken that the two solutions are well mixed.

A precipitate is often caused to appear more rapidly by mixing or agitating the liquid, or by warming it.

Solid substances are occasionally used to produce precipitates. Thus a metal is not unfrequently precipitated from the solution of its salt by the immersion in the liquid of another metal.

Expt. 81.—Dip a clean penknife-blade into some of the copper sulphate solution (Expt. 74), to which a few drops of dilute sulphuric acid have been added. After a short time metallic copper will be precipitated from the solution and will cover the iron as a red film.

The appearance and character of a precipitate should be carefully noted, since the precipitate is frequently identified by these qualities. It may be coloured, flocculent, gelatinous, light or heavy, and crystalline or non-crystalline. The reagent should also be added in gradually increasing quantity, in order to ascertain whether the precipitate varies in character with varying proportions of the reagent, and whether the precipitate is soluble in excess of the reagent.

Expt. 82.—Note down full particulars concerning the appearance and character of the precipitates which are produced when the following liquids are mixed:—

Solutions of ammonium hydrate and of alum. Dilute hydrochloric acid and silver nitrate solution. Solutions of potassium chromate and lead acetate. Solutions of hydrogen sulphide and mercuric chloride. Solutions of potassium iodide and mercuric chloride. Solutions of potassium hydrate and alum.

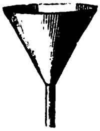
FILTRATION.

70. Filtration.—This process is generally used to separate a solid substance from a liquid. It serves to separate a soluble from an insoluble substance after the mixed substances have been treated with a suitable solvent; it is also constantly employed to effect the separation of a precipitate from a liquid in which it is suspended.

The liquid containing the solid is poured upon porous paper, called *filter-paper*. The suitably folded paper is known as the *filter*. The liquid itself passes through the pores of the paper, but the solid particles of the precipitate are retained upon the surface of the paper. The liquid which has passed through the paper is termed the *filtrate*. A filtrate may frequently be coloured by some substance *dissolved* in it, but it must always be perfectly free from turbidity caused by solid particles *suspended* in it.

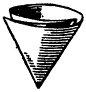
EXPT. 83.—Measure a glass funnel along its sloping side from shoulder to rim (fig. 42). Select a circular filter-paper, the radius of which is somewhat less in length than this measure; fold the paper across into a semicircle, and then again at right angles into a quadrant; then open out the quadrant into the little conical bag (fig. 43).

FIG. 42.



THE FUNNEL.

FIG. 43.



THE FILTER.

Now press the folded filter into the *dry* funnel, and alter the folding if necessary until the paper fits the glass closely. Then moisten it all over with water from the wash-bottle. The filter is now ready for use.

The funnel may be placed with its neck in a test-tube or flask while the filter is being used. Care must, however, be taken that there is a space between the neck of the funnel and the inside of the tube, since the filtration is stopped if the egress of air is prevented. The funnel is often preferably supported upon the ring of a wooden filter-stand (fig. 44), and the filtrate is received in a small beaker instead of in a test-tube.

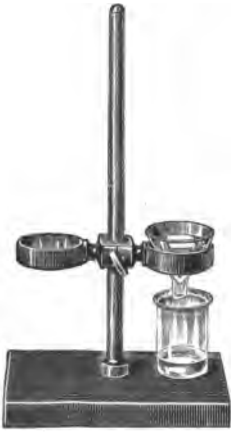
The edge of the filter must always be below the rim of the funnel, and the liquid inside the funnel must not reach the upper edge of the filter-paper.

Expt. 84.—Grind some potassium nitrate and some fine silver sand together in a mortar until the substances are intimately mixed. Pour a little water upon the mixture and mix it well with the pestle. Then transfer the liquid with the solid to a filter. The sand will remain upon the filter, and the solution of potassium nitrate will pass through as a clear liquid. The potassium nitrate, which has thus been separated in solution from the sand, may now be recovered in the solid state by evaporating the filtrate.

Expt. 85.—Filter the liquid which contains the precipitate of barium carbonate from Expt. 80. The filtrate should run through perfectly clear, leaving the barium carbonate upon the filter. Keep the funnel with the filter and the precipitate.

Sometimes the filtrate passes through the filter in a turbid condition. If this is the case the whole of the filtrate, or the first portions of it, must be poured once or twice through the same filter until the turbidity is removed. The process may be hastened by pouring the liquid through two filter-papers folded together so as to form one filter; a double filtration is thus effected in one operation.

Fig. 44.



The Filter-stand.

It should be borne in mind that the rate at which a liquid passes through the filter is increased by heating the liquid. Hence a solution should always be heated before it is filtered, unless this is inadmissible for special reasons.

DECANTATION.

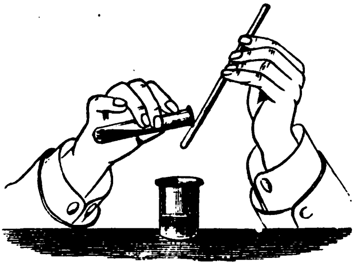
71.—**Decantation.**—A precipitate which settles down rapidly may frequently be separated from the liquid by the process of decantation without the use of a filter.

In order to effect this, the vessel which contains the liquid and the precipitate is allowed to stand at rest until the precipitate has settled. The liquid is then carefully poured off or *decanted* by gently inclining the vessel.

A wetted glass rod should be pressed against the edge or lip of the vessel (fig. 45), in order to prevent the disturbance of the precipitate during the decantation of the liquid.

If the process is carefully performed it will effect a nearly complete separation of a heavy precipitate from the liquid.

FIG. 45.



DECANTATION OF LIQUID.

EXPT. 86.—Add some sodium carbonate solution to a hot solution of lead acetate. A heavy precipitate of lead carbonate will be formed. This may be readily separated from the liquid by decantation in the manner just described.

SECTION VI.

CARBON, OXIDES OF CARBON.
CARBONATES.
COAL-GAS, METHANE, ETHYLENE.
COMBUSTION, FLAME.

CARBON.

72. **Carbon** occurs in several different forms in nature. The diamond is carbon in a crystalline and almost pure condition; graphite is another crystalline and generally less pure form of carbon; while the different varieties of coal consist of carbon in a condition usually far less pure.

Charcoal and coke are impure and artificial forms of carbon, which are made from wood and from coal respectively by heating them out of contact with air. The volatile matter is thus driven off, and a residue of carbon is left, which contains any mineral matter which was originally present in the wood or coal.

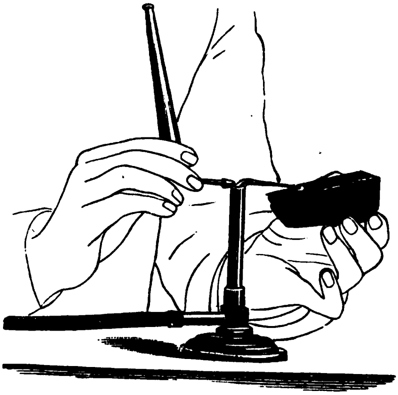
EXPT. 87.—Heat a small piece of wood in a test-tube, held in a horizontal position. At first water vapour will be given off, and will condense in the upper part of the tube. Afterwards inflammable gases will be evolved, and may be kindled at the mouth of the tube. Later on, dark brown tarry vapours pass off and collect in the liquid form inside the tube. When the tarry matter ceases to be given off, turn out the solid residue from the bottom of the tube; this will consist of a piece of black wood-charcoal.

EXPT. 88.—Heat a small piece of coal in a test-tube in a similar way. Gas will at first be given off, which may be kindled and burnt with a luminous flame at the mouth of the tube. After a time, black tarry matter with an offensive smell will collect in the upper part of the tube. As soon as the tar has ceased to come off, turn out the solid residue from the tube; and notice that it is ordinary coke.

73. **Reduction by Charcoal.**—Strongly-heated carbon is used as a *reducing agent* for withdrawing oxygen from a substance, and the smelting of metals from their ores is frequently effected by its means. Iron, copper, tin, and zinc may be *reduced* from their oxides and smelted from their ores by means of carbon at a high temperature.

Expt. 89.—Make a small shallow hole on the surface of a piece of charcoal by means of a knife, and place a small quantity of lead oxide in the cavity. Heat the lead oxide in the blowpipe-flame (23) in the manner shown in fig. 46.

Fig. 46.



Heating on Charcoal.

The oxygen of the lead oxide will combine with the carbon of the charcoal, and a bright globule of metallic lead will be left in the cavity.

74. **Decolorising Power of Charcoal.**—Charcoal possesses the power of destroying many coloured substances in solu-

tion. This is due in some cases to an absorption of the colouring matter in the charcoal and in others to the power possessed by the carbon of absorbing oxygen from the air which oxidises the coloured substance. Charcoal made from bones seems to act most rapidly.

EXPT. 90.—Warm a little blue litmus solution with a fair quantity of bone charcoal. Allow the mixture to stand a short time and then filter it. The blue colour will be found to have disappeared, and a colourless liquid will remain.

OXIDES OF CARBON.

When carbon is burnt in a large supply of oxygen, it is converted into carbon dioxide gas (Expt. 49, p. 46). But if the supply of oxygen is limited, part of the carbon will combine with a less proportion of oxygen, and some carbon monoxide gas will be produced.

Experiments which illustrate the preparation and the study of the properties of these two gases are given below.

CARBON MONOXIDE GAS, CO.

Carbon monoxide is produced when a limited volume of air is passed over a large surface of red-hot charcoal: $C+O=CO$. The gas is often formed in this way in open grates, and is seen burning with its characteristic blue flame at the upper surface of the glowing fuel. It is poisonous when it is inhaled in any quantity.

Two methods for preparing carbon monoxide are described below (75, 76). The gas is most easily made by heating sodium formate with sulphuric acid (76). The preparation from oxalic acid (75) is, however, also described, because it furnishes a good illustration of the ordinary method of purifying a gas by washing it with a suitable liquid, or by passing it over fragments of a suitable solid.

75. Preparation of Carbon monoxide from Oxalic acid.—A mixture of carbon monoxide with an equal measure of carbon dioxide may be prepared by heating crystals of oxalic acid with strong sulphuric acid:

$$H_2C_2O_4.2H_2O+H_2SO_4=CO+CO_2+H_2SO_4.3H_2O.$$

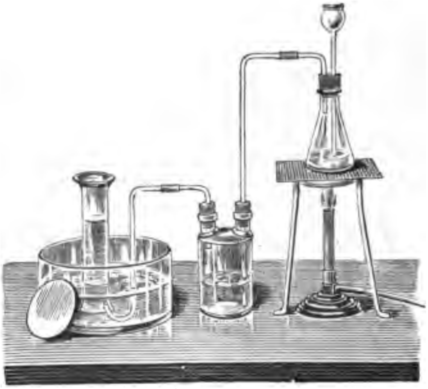
EXPT. 91.—Pour upon a few crystals of oxalic acid, contained in a boiling-tube, about twice their volume of strong sulphuric acid, and heat the mixture. In a short time effervescence will commence, showing that gas is being evolved.

Introduce into the mouth of the boiling-tube a glass rod, which has been freshly dipped into lime-water; the drop of lime-water hanging upon its end will become milky, proving that carbon dioxide is present in the escaping gas. Hold the mouth of the boiling-tube against a Bunsen-flame, a blue flame will appear at the mouth of the tube, showing that carbon monoxide gas is also being evolved.

In order to remove the carbon dioxide and to obtain pure carbon monoxide, the mixture of gases is either *washed* with a solution of caustic soda, or it is passed over pieces of solid caustic soda, or of soda-lime. Caustic soda absorbs the carbon dioxide readily, but it does not affect carbon monoxide and allows it to pass on:

$$CO + CO_2 + 2NaHO = CO + Na_2CO_3 + H_2O.$$

FIG. 47.



PREPARATION AND COLLECTION OF CARBON MONOXIDE.

EXPT. 92.—Heat a mixture of oxalic acid crystals and strong sulphuric acid in a flask fitted as is shown in figure 47. Remove the flame, and moderate the heat, as soon as effervescence commences, else the acid will froth over.

Allow the gases which are evolved to pass through a wash-bottle containing caustic soda solution, or through a suitable tube filled with fragments of soda-lime, or with fragments of pumice-stone moistened with strong caustic soda solution. The bulbed tube (*a*) or the U-tube (*b*), shown in fig. 36 (p. 58), are commonly employed as receptacles for the solid reagents. If the carbon dioxide is to be completely removed, the gases must be passed through two or more such tubes or bottles, and the stream of gas must be slow.

The carbon monoxide gas may be collected over water. It will produce no milkiness when it is shaken with lime-water, unless the stream of gas has been so rapid that the caustic soda has not been able to absorb the carbon dioxide completely.

76. **Preparation of Carbon monoxide from Sodium formate.**—When sodium formate is heated with strong sulphuric acid, carbon monoxide alone is given off:

$$NaHCO_2 + H_2SO_4 = CO + NaHSO_4.H_2O.$$

Carbon monoxide is obtained free from carbon dioxide by this process. The preparation may be carried out in the apparatus shown in fig. 29 (p. 41).

77. **Carbon monoxide burns in Air, forming Carbon dioxide.**—Carbon monoxide resembles hydrogen by being inflammable and by extinguishing flame. It also burns explosively when its mixture with a suitable proportion of oxygen or of air is kindled. Hence, before this gas is collected in any quantity, a test-tube should be filled with it, and this small quantity of gas should be kindled in order to see that it does not burn explosively owing to admixture with air.

Expt. 93.—Push a burning taper up into an inverted jar filled with carbon monoxide. The gas will burn with a blue flame at the mouth of the jar, but the flame of the taper will be extinguished.

As soon as the gas has ceased to burn inside the cylinder, pour in a little lime-water and shake it about. The liquid will become milky, showing that the combustion of carbon monoxide in the oxygen of the air has produced carbon dioxide gas: $CO + O = CO_2$.

78. **Test for Carbon monoxide.**—Carbon monoxide is recognised by burning with a pale blue flame in the air, and producing carbon dioxide which renders lime-water milky.

CARBON DIOXIDE GAS, CO_2.

79. This gas was formerly called carbonic acid, a name which is incorrect, since all acids contain hydrogen.

When carbon was burned in oxygen (Expt. 49, p. 46) carbon dioxide gas remained in the jar. The gas is much more easily obtained by pouring hydrochloric acid upon some pieces of marble:

$$CaCO_3 + 2HCl = CO_2 + H_2O + CaCl_2.$$

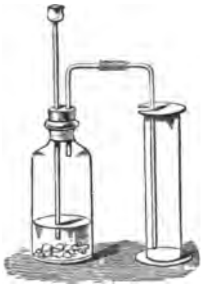
Expt. 94.—Rinse out the apparatus which was used for preparing hydrogen (Expt. 58, p. 55), or fit the apparatus shown in fig. 48, and place in it some small pieces of marble. Fit into the rubber joint a delivery-tube bent at right angles, as is shown in fig. 48. Pour in sufficient water to cover the marble and the end of the funnel-tube, and add strong *commercial* hydrochloric acid through the funnel-tube until the gas comes off with brisk effervescence.

Since this gas is soluble in water and is heavier than air, it should be collected by downward displacement. For this purpose the delivery-tube is placed in a jar with its end nearly touching the bottom, and the gas is allowed to pass into the jar for several minutes.

It is well to check diffusion by covering the mouth of the jar with a small disc of cardboard, which has had a slit or hole cut in it for the delivery-tube.

Carbon dioxide will soon fill the jar, and the jar is known to be full when the flame of a lighted paper held inside its mouth is extinguished.

Fig. 48.



Preparation and Collection of Carbon Dioxide.

80. Properties of Carbon dioxide.—Carbon dioxide may be proved to be heavier than air, by showing that it remains in a vessel which is open above, and falls out of one which is open below.

Expt. 95.—Allow a jar of carbon dioxide to stand uncovered with its mouth upwards for a few minutes. Then place in the jar a burning taper. The carbon dioxide will be proved to be still present in the vessel by the immediate extinction of the flame. Now hold the jar for several minutes with its mouth downwards, and introduce a lighted taper; the flame will continue to burn as it does in air, showing that only air is present in the vessel.

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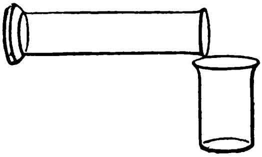
Expt. 96.—Tilt a jar of carbon dioxide a little beyond the horizontal position (fig. 49), with its mouth over that of a small beaker. After a short time, it may be shown by means of a lighted taper that the gas has left the jar and is present in the beaker.

Expt. 97.—Pour some lime-water from a test-tube into a jar of carbon dioxide, and shake the liquid round in the jar: the lime-water will at once become milky. The lime, which was dissolved in the water and was therefore invisible, has been converted by the carbon dioxide into chalk or calcium carbonate; and this, being an insoluble substance, remains suspended as a white *precipitate* in the water: $CaH_2O_2 + CO_2 = CaCO_3 + H_2O$.

Fig. 49.



Pouring off Carbon dioxide.

Carbon dioxide gas readily dissolves in cold water, forming a liquid which contains carbonic acid. Its solubility in water, and its removal by boiling, may be shown by the following experiments.

Expt. 98.—Displace the air by means of carbon dioxide from a jar already half filled with cold water. Then close the mouth of the jar tightly with the wetted palm of the hand, and shake the gas and the water together vigorously for a short time. The mouth of the jar will now firmly adhere to the hand, owing to the reduction of the internal pressure caused by the absorption of the gas.

A further proof of the solution of the gas is afforded by adding lime-water to the liquid; the lime-water will immediately become milky.

Expt. 99.—Dip the end of the delivery-tube of the generating apparatus (fig. 48) into some water, which has been coloured blue with litmus and poured into a small beaker. The blue litmus will become dull red in colour, showing that the water has become acid. Boil half of this solution for a short time. The litmus will change back to blue, showing that the gas is readily expelled from its solution in water by boiling.

Expt. 100.—Dilute some lime-water in a small beaker with an equal quantity of distilled water, and allow carbon dioxide gas to bubble through it. The milkiness which is produced at first will gradually disappear, showing that calcium carbonate is soluble in carbonic acid.

Boil some of this clear liquid for several minutes. The carbonic acid will be removed, and the liquid will become milky again, because the chalk can no longer remain dissolved. Now pour out the water: part of the calcium carbonate will be seen adhering to the inside of the vessel; it may be readily removed by rinsing the vessel with a little hydrochloric acid.

The above experiment explains the origin of the coating or *incrustation* inside kettles and steam boilers, in which hard chalk water has been boiled. Such water contains chalk dissolved by carbonic acid; this acid is destroyed by the removal of the carbon dioxide when the water is boiled, and a great part of the chalk is deposited upon the inside of the vessel.

81. Tests for Carbon dioxide.—The presence of carbon dioxide gas may be detected by its power of extinguishing a burning taper, and of turning lime-water milky. The latter test distinguishes it from nitrogen.

These tests may now be employed to prove that carbon dioxide is evolved from the lungs during the process of respiration, and also that it is produced by an ordinary flame.

Expt. 101.—Breathe through a glass tube into lime-water contained in a small beaker. The lime-water will become milky.

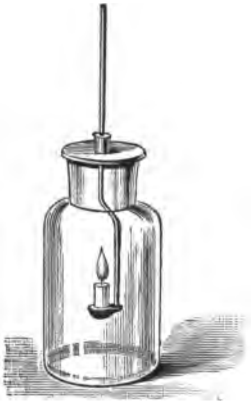
Expt. 102.—Light a small piece of candle or wax-taper upon a deflagrating spoon, and introduce it into a bottle of air (fig. 50). After the flame has burnt for a short time it will be extinguished. Now pour in some lime-water and shake it round in the bottle: the liquid will become milky.

Carbon dioxide gas is constantly being introduced into the atmosphere by respiration and by combustion. It is not ordinarily present in sufficient quantity to extinguish a burning taper; but its presence may be shown by means of lime-water (Expt. 56, p. 53).

THE CARBONATES.

82. Carbonic acid and Carbonates.—When carbon dioxide gas is passed into litmus solution (Expt. 99, p. 81) the litmus solution becomes red, showing that an acid has been produced. This acid is very unstable, and is decomposed when its solution is heated. The salts which correspond to this acid are called carbonates; they are much more stable than carbonic acid, but they are easily decomposed by the action of acids.

FIG. 50.



PRODUCTION OF CARBON DIOXIDE BY CANDLE FLAME.

83. Sodium carbonate.—The preparation of sodium carbonate may be taken as typical of the method of preparing the metallic carbonates. Many of them may be prepared by passing carbon dioxide through a solution of the oxide of the metal.

EXPT. 103.—Pass carbon dioxide gas through 50 c.c. of sodium hydrate solution until a drop of the liquid, taken out on the end of a glass rod, no longer turns red litmus paper blue. Evaporate (66) the solution to about one quarter its volume, and place it on one side to crystallise. Transparent crystals of sodium carbonate will form. They have a composition denoted by the formula $Na_2CO_3.10H_2O$.

84. Test for Carbonate.—When hydrochloric acid, or almost any other acid, is added to a carbonate, carbon dioxide gas is given off with effervescence. This gas may be recognised by its property of turning lime-water milky.

Expt. 104.—Place a little solid sodium carbonate in a test-tube and add dilute hydrochloric acid. The heavy carbon dioxide gas will remain in the tube. Gradually slope the tube so as to pour out the gas into another test-tube containing a little lime-water, without allowing any of the liquid to flow out. On closing this tube with the thumb and shaking, the lime-water will become milky.

85. Loss of Weight by a Carbonate when it is heated.—Many carbonates give off carbon dioxide gas when they are heated, and leave a residue of oxide of the metal. Calcium carbonate gives off the whole of its carbon dioxide when it is heated to bright redness for about fifteen minutes.

Expt. 105.—Place a little pure precipitated calcium carbonate in a piece of combustion-tube six inches long and closed at one end. Fit a cork with a bent delivery-tube into the open end, and allow the end of the delivery-tube to dip into a little lime-water contained in a test-tube. Now heat the calcium carbonate to redness. Bubbles will pass through the lime-water, and after a short time the liquid will become milky, showing that carbon dioxide has been evolved.

If the heating is continued for some time the whole of the carbon dioxide will be expelled from the carbonate. This may be proved by heating some more of the carbonate on platinum-foil to redness for some time, then moistening the mass with water, and adding a few drops of dilute acid. No effervescence will take place, showing that the whole of the carbon dioxide has been driven off.

In the above experiment the calcium carbonate has been decomposed. Carbon dioxide has been expelled, and quicklime or calcium oxide has been left.

EXPT. 106.—Make a square box of platinum-foil about 1·5 inches in the side and 0·25 inch deep. This may be effected by folding a piece of the foil two inches square over the end of a piece of wood 1·5 inch square.*

Weigh the box on the balance, and weigh into it one gram of pure dried precipitated calcium carbonate.

Place the box on a pipeclay triangle supported on a retort-stand over the large blowpipe, and heat it for about ten minutes. Then allow the box to cool, and weigh it. Now heat it for five minutes more and weigh it again, and continue the alternate heating and weighing until two successive weighings give identical results.

The residue on the foil is calcium oxide, and the loss of weight observed is the weight of the carbon dioxide which was present in the one gram of calcium carbonate. Calculate from this result the weight of CO_2 in 100 grams of the carbonate.

86. Measurement of the Volume of Carbon dioxide evolved by an Acid from Calcium carbonate.

EXPT. 107.—The apparatus described in Expt. 65 (p. 60) is employed, but since CO_2 is somewhat soluble in water, it is necessary to saturate the water in the aspirator with the gas, before making the estimation. This may be effected by preparing CO_2 in the way already described in Expt. 94 (p. 80), and passing the gas through the water for about twenty minutes.

Weigh out into a four-ounce flask one gram of iceland-spar or of marble. Fit up the aspirator in the same way as in Expt. 65 (page 60), and pour the water which has been saturated with CO_2 into the bottle.

Remove the cork from the flask, pour in about 14 c.c. of water, and fill the small tube with strong hydrochloric acid.

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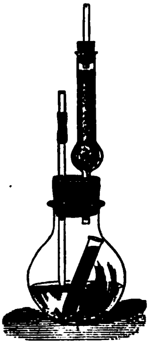
* A porcelain crucible may be used instead of a platinum box, but it requires to be heated longer than the platinum does. If a porcelain crucible is used, it should be heated for about half an hour in a muffle furnace.

Now replace the cork and open the clamp on the rubber tube. Allow the acid to act upon the carbonate, measure the volume of water displaced by the gas, and correct the volume of the gas, thus obtained, for temperature, as is explained in the Note on page 50.

Now determine in a precisely similar way the volume of gas which is evolved when nitric acid is used instead of hydrochloric acid. The volume of gas evolved by nitric acid from one gram of calcium carbonate should be exactly equal to that evolved by hydrochloric acid, showing that the same amounts of CO_2 are evolved by different acids.

87. Estimation of the Percentage Weight of Carbon dioxide in Calcium carbonate.—When hydrochloric acid acts upon calcium carbonate, carbon dioxide is set free and is evolved as gas. The decomposition may be carried out in such a way that no loss of material takes place except that due to the evolution of the carbon dioxide. Under these circumstances the percentage weight of carbon dioxide in a known weight of the calcium carbonate will be equal to the loss of weight which a mixture of the carbonate with excess of acid suffers by complete decomposition.

FIG. 51.



ESTIMATION OF CO_2 IN CALCIUM CARBONATE.

EXPT. 108.—Close a six-ounce flask with a doubly perforated, sound cork. Through one of the perforations pass a calcium chloride drying tube (fig. 36 *a*, page 58). Through the other pass a narrow tube, the lower end of which is contracted and the upper end closed with rubber tube, stopped with a piece of glass rod. Ascertain that the apparatus is air-tight. Weigh out 0·75 gram of pure iceland-spar; introduce it into the flask and pour in water until the substance is just covered.

Now nearly fill a small test-tube, three inches in length by half an inch in diameter, with strong hydrochloric acid, and place it in the flask. Then weigh the whole apparatus.

Next gently tilt the flask so as to allow the acid to flow little by little out of the test-tube, and to come into contact with the carbonate, producing a slow and regular effervescence.

As soon as the solid has dissolved and the effervescence has ceased, push down the glass tube so that the point just touches the surface of the liquid in the flask. Then open the rubber tube by removing the glass stopper, and warm the contents of the flask by placing the apparatus on the water-bath or by heating it *gently* by means of a small flame. At the same time draw a *slow* current of air through the flask for several minutes by applying gentle suction at the free end of the drying-tube. The calcium chloride in this tube serves to dry the carbon dioxide as it leaves the apparatus, and thus prevents moisture from escaping with the gas.

Then replace the glass plug, allow the flask to cool, and weigh the apparatus again.

The difference between the weights of the apparatus before and after the experiment gives the weight of the carbon dioxide which has been evolved during the reaction.

From this weight, and from that of the calc-spar used, the percentage of carbon dioxide in the calc-spar can be calculated. This should correspond to the proportion which has been found by igniting the carbonate; since the action both of an acid and of a high temperature removes the whole of the carbon dioxide from the carbonate.

COMBUSTION.

88. It has been already shown (37) that chemical action takes place when certain substances are heated in the air. This action is due to the combination of the substance as a whole, or of some of its constituents, with the oxygen of the air. The process is known under the general name of oxidation; but if it is attended with the production of heat and light, it is usually called combustion.

The elements carbon and hydrogen are constituents of ordinary *combustible* materials, such as coal, wax, oil, and the various kinds of illuminating gas and vapour. Coal-gas is a convenient substance for use in studying some of the phenomena of combustion.

COAL-GAS AND ITS CONSTITUENTS.

89. Coal-gas is prepared by heating coal out of contact with air. It is made on a large scale by heating coal in clay or iron vessels called retorts. Under these conditions other bodies besides illuminating gas are formed, such as ammonia, carbon dioxide, and tar: these are carefully removed from the gas before it is used.

A small quantity of coal-gas may be prepared the following way.

EXPT. 109.—Break up some coal into small fragments and introduce it into an ordinary clay tobacco pipe, until the bowl is almost filled.

Finely powder some Stourbridge clay and make it into a stiff paste with water. Press this clay into the upper part of the bowl of the pipe until it is closed, and then heat the bowl in the steam-oven until the clay is dry and hard.

Now support the pipe securely on the ring of a retort-stand, and heat the bowl in the Bunsen-flame.

In a short time gas, accompanied by tar vapour, will be seen to issue from the stem of the pipe; this gas may be kindled, and will burn with a luminous flame.

90. Constituents of Coal-gas.—The principal constituents of coal-gas may be classified in two groups.

One group includes those gases which during their combustion are mainly producers of heat: these are hydrogen, carbon monoxide, and methane. The other group includes those which produce both heat and light when they are being burnt, such as ethylene gas and acetylene.

Hydrogen and carbon monoxide have been already described. Experiments with methane, ethylene, and acetylene are described below.

METHANE GAS, OR MARSH GAS.

91. Preparation of Methane from Sodium acetate.—This gas may be prepared by heating an intimate mixture of dried sodium acetate with soda-lime:

$$NaC_2H_3O_2 + NaOH = CH_4 + Na_2CO_3.$$

EXPT. 110.—Heat some crystallised sodium acetate in a shallow iron sand-bath over the Bunsen-flame, until steam is no longer given off. The fused salt will set into a white mass as it cools. Mix one part of this dry salt with four parts of dry soda-lime, then reduce the mixture to fine powder, and heat the powder to redness in an apparatus such as that shown in fig. 31 (p. 45). In this apparatus it is necessary to substitute a hard-glass tube for the test-tube, since an ordinary test-tube would be softened by the heat which must be applied for the above decomposition. As soon as the air has been expelled from the apparatus, fill two cylinders with methane over water in the usual way, and fill another dry cylinder by direct displacement of the air by the light gas.

92. Properties of Methane.—Burn the methane in one of the wet jars. The flame will be found to be only slightly luminous. Burn the gas in the jar which was filled by upward displacement; the sides of the jar will become dimmed with moisture, proving that one of the products of the combustion is water. Pour a little lime-water into the jar: the lime-water will become milky, showing that carbon dioxide is also formed by the combustion.

ETHENE, OR ETHYLENE GAS.

93. Preparation of Ethene from Alcohol.—Ethene may be prepared by heating alcohol with strong sulphuric acid:

$$C_2H_6O + H_2SO_4 = C_2H_4 + H_2SO_4.H_2O.$$

EXPT. 111.—The apparatus shown in fig. 47 (p. 78) may be used. The flask should, however, be round and not conical in shape, and must not be less than 14 ounces in capacity, since the preparation-mixture froths considerably.

A mixture of one volume of alcohol, of 0·83 specific gravity, with five volumes of strong sulphuric acid, is made by shaking the liquids together in the generating-flask. This mixture must not be more than half an inch in depth in the flask. A solution of sodium hydrate is placed in the two-necked washing-bottle, and serves to absorb the sulphur dioxide which escapes during the preparation.

The mixture in the generating-flask is heated carefully with constant watching, since it is apt to froth.

Ethene gas, when mixed with air, burns explosively, and care must therefore be taken to expel the air from the preparation vessels completely before any quantity of the gas is collected and burnt. Two cylinders should be filled with the gas by collecting it over water, and another dry jar should be filled by direct displacement of the air.

94. **Properties of Ethene.**—Ethene may be recognised by the character and products of its flame, and by its power of combining with bromine and destroying its colour.

Expt. 112.—Burn ethene in one of the wet jars and note its *very* luminous flame. Burn also the gas contained in the dry jar and note that the sides of the jar become dimmed with moisture. Close the jar in which the gas has been burnt with a glass plate as soon as the combustion is ended, and shake up the products of combustion with lime-water. The lime-water will become milky, proving that carbon dioxide is also a product of the combustion of ethene.

ACETYLENE.

95. **Preparation of Acetylene from Calcium carbide.**—When calcium carbide is dropped into water, acetylene gas is rapidly given off:

$$CaC_2 + H_2O = C_2H_2 + CaO.$$

Expt. 113.—Fill a small cylinder or a boiling tube with water; close the mouth and invert it, mouth downwards, in a vessel of water, so as to retain the water in the cylinder. Drop into the vessel a piece of calcium carbide about the size of a hazel-nut, and collect the gas which is generated by shifting the tube until its mouth is over the piece of calcium carbide.

When the tube is full of gas remove it from the water and ignite the gas. It will burn with a very luminous smoky flame.

FLAME.

96. In order that a burning substance may produce flame, it is necessary that it shall be in the condition of vapour or

gas. The area of chemical action and of heat in a flame is the surface of contact of the gas or vapour with the air. Hence under ordinary conditions all flames are hollow. This may be readily shown to be true of a candle flame.

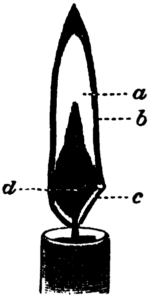
Expt. 114.—**A Candle flame is hollow.**—Light a candle, and when the flame is fully developed press upon it for a second a piece of white paper, causing the paper just to touch the top of the wick. When the paper is withdrawn a more or less perfectly-formed charred ring with a white centre will be seen upon the upper surface of the paper. This shows that no combustion or production of heat takes place in the middle of the flame, but that where the paper cuts through the conical exterior much heat is being produced.

Expt. 115.—**Structure of a Candle flame.**—Carefully examine the candle flame. Three zones will be noticed.

(1) *An inner dark zone*, which contains the inflammable vapour emitted by the wick. Hold a short piece of glass tube, which has just been heated in the Bunsen-flame, in an inclined position, with its lower end in the dark portion of the candle flame. White vapour will be seen escaping from the tube. This vapour may be kindled by the flame of a taper, showing that this portion of the flame consists of inflammable vapour.

(2) *A luminous mantle.*—Hold the end of a glass rod in the bright part of the flame; it will be instantly coated with soot. The light which is emitted by this part of the flame is due to the presence in it of white-hot carbon particles which have been formed from the inner inflammable vapour, and in this portion of the flame combustion is therefore far from being complete.

Fig. 52.



Candle flame.

(3) A very pale outer mantle may be seen if the edge of the flame is carefully examined, while the bright part is shielded from the eye. This mantle becomes coloured yellow if a little dry sodium carbonate is scattered above the flame, and it is then easily seen.

In this portion of the flame final and complete combustion of the constituents of the candle is taking place, and no soot is therefore deposited upon a cold rod held in the edge of the flame. Water and carbon dioxide may be shown to be products of the combustion by inverting a clean dry glass cylinder over the flame. The cylinder will become dimmed inside with moisture; and if the cylinder is closed with a glass plate as soon as the candle flame becomes extinguished, a little lime-water poured in and shaken round will become milky.

97. **The Bunsen-flame and other Similar flames** are produced by burning gas which has been mixed with air before it is burnt. The luminosity of the flame is thus almost abolished, and accordingly the flame deposits no soot upon a cold surface.

The following experiments will illustrate the principles upon which flames of this kind are produced.

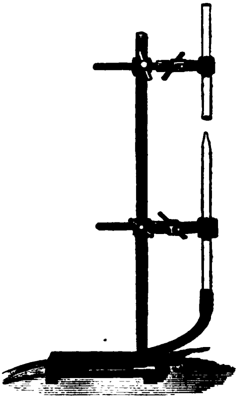
Expt. 116.—Draw out a piece of glass tube to a jet at one end. Support the tube vertically in a clamp, and attach the lower end to the gas supply by means of a rubber tube, as is shown in fig. 53.

Now turn on the gas and kindle it at the jet. It will burn with a luminous flame.

Extinguish the flame and pass down over the jet a piece of glass tube about six inches long and of somewhat larger bore than the jet-tube. Arrange the jet-tube so that the gas will pass up the centre of the larger tube. Now turn on the gas again and kindle it at the top of the broader tube. If this tube has been properly arranged and the supply of gas suitably adjusted the gas will burn with a feebly luminous flame, which will deposit no soot upon a cold body.

If a smoking piece of paper is brought near to the jet, it will be seen that air is drawn in at the bottom of the broader tube by the up-rush of gas from the jet. This mixes with the gas in the tube, and accordingly a mixture of gas and air is burnt at the top of the tube.

FIG. 53.



PRODUCTION OF A BUNSEN-FLAME.

Compare the construction of the Bunsen-burner with the above apparatus.

EXPT. 117.—Extinguish the flame and replace the upper tube by a piece of fine wire gauze held horizontally about two inches above the jet. Now kindle the gas above the gauze. The flame will be non-luminous and similar to that already produced by means of the broader tube. In this case the mixture of air with the gas takes place between the jet and the gauze. The flame above the gauze does not descend to the jet, because the large cooling surface of the gauze cools the flame in contact with it below the temperature of ignition of the gaseous mixture.

98. **The Davy lamp.**—The above experiment illustrates the principle of the Davy lamp. The gauze which surrounds the oil flame of

the lamp prevents the ignition of an inflammable mixture outside the gauze, although the mixture has been kindled inside the gauze by contact with the oil flame.

Expt. 118.—Place a small candle on a block of wood, kindle the wick, and surround the flame with a sheet of fine wire gauze which has been bent into a cylinder. Cover the top of the cylinder with a small piece of gauze so as to surround the candle completely. Now let the gas from a Bunsen-burner play against the gauze. The gas will be kindled *inside* the gauze cylinder, but it will not take fire outside unless some openings have been left unguarded by the gauze in constructing the gauze envelope.

SECTION VII.

THE HALOGENS.
CHLORINE, HYDROCHLORIC ACID.
CHLORIDES. ACIDS AND SALTS.

THE HALOGENS.

99. The group of elements known as the halogens, or salt-producers, contains four elements, fluorine, chlorine, bromine, and iodine. These possess many properties in common. Fluorine is very difficult to prepare. A small quantity of the other elements may be made from their compounds, in order to study their properties.

EXPT. 119.—**Chlorine** is readily made in the following manner. Powder a little solid sodium chloride, and mix it intimately with four times as much powdered manganese dioxide. Place the mixture in a boiling-tube, add a little strong sulphuric acid, and warm the mixture gently. Chlorine gas will be slowly given off, and will fill the upper part of the tube.

$$2NaCl + MnO_2 + 3H_2SO_4 = Cl_2 + MnSO_4 + 2NaHSO_4 + 2H_2O.$$

Notice the pale yellowish-green colour of the gas, and its suffocating smell. Hold a piece of moistened red litmus paper in the gas and notice that it is bleached immediately.

EXPT. 120.—**Bromine** may be made in similar manner by substituting sodium bromide for sodium chloride. When the mixture is warmed, a reddish-brown vapour with a very unpleasant and suffocating smell will fill the upper part of the tube. It will be found to possess a slight bleaching power upon moistened red litmus.

If a glass rod, which has been previously dipped into moistened starch, is held in this vapour, the starch will be stained orange-red in colour.

This bromine vapour may be condensed by suitable means into a heavy reddish-brown liquid.

EXPT. 121.—**Iodine** may be made by heating a mixture of potassium iodide and manganese oxide with strong sulphuric acid. A purple vapour will be formed in the lower part of the tube, and will condense to a blue-black solid in the upper part.

EXPT. 122.—The colour of the vapour may be seen more distinctly by heating a fragment of iodine in a test-tube. A beautiful violet vapour will be evolved, which will produce sparkling bluish-black crystals in the cold part of the tube. The colour of red litmus paper will be destroyed by iodine vapour, but the paper will be stained yellow by the iodine.

EXPT. 123.—Starch assumes a deep blue colour when it is brought into contact with iodine. This change of colour is easily produced in the following way. Warm a small crystal of iodine in a little water. Add this liquid to some cold dilute starch solution, which has been freshly made by boiling a little starch in water. The starch solution will at once assume a deep blue colour.

From the above experiments it will be seen that each of the properties of the halogens is gradationally developed in the halogens. This will become evident by tabulating (1) the bleaching effect, (2) the colour of the vapour, and (3) the colour effect on starch of the three halogens, as these properties have been illustrated above.

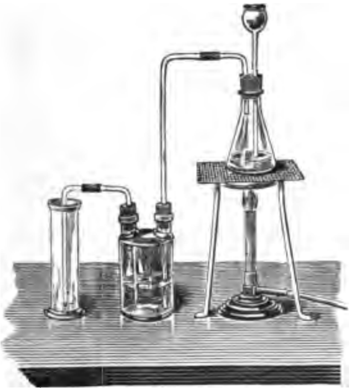
CHLORINE.

100. Chlorine is the most abundant of the halogen elements. It may be prepared for experimental purposes by heating manganese dioxide with hydrochloric acid.

The gas may be made in the apparatus shown in fig. 54. The washing-bottle may either be dispensed with, or it may be partly filled with water in order to free the chlorine from hydrochloric acid gas.

Expt. 124.—Place some small pieces of manganese dioxide in the flask. Pour in a mixture of strong hydrochloric acid with about one-third its measure of water; and heat the mixture gently in a draught-closet. A greenish-yellow gas will be evolved, which may be collected by downward displacement since it is much heavier than air:

Fig. 54.



Preparation of Chlorine.

$$MnO_2 + 4HCl = Cl_2 + MnCl_2 + 2H_2O.$$

This gas has a very destructive action on the lungs, and must on no account be inhaled in any quantity.

101. Recognition of Chlorine.—Chlorine is usually recognised by its yellowish-green colour, by its peculiar smell, and by its power of bleaching moist vegetable colours.

Expt. 125.—The bleaching power may be shown by introducing a piece of moistened litmus paper, or of *Turkey-*

red fabric into a jar of the gas. The colour will be destroyed.

When a burning wax taper is plunged into a jar of chlorine, it continues to burn with a very smoky flame. If hot turpentine, soaked up by a strip of filter-paper, is introduced into chlorine, it catches fire spontaneously, and gives rise to dense smoke. A piece of dutch-foil or copper-leaf also burns when it is dropped into chlorine.

Water will be found to dissolve chlorine gas, and to acquire its peculiar colour and properties. The solution may be prepared by causing the gas to bubble through the water.

HYDROCHLORIC ACID, OR HYDROGEN CHLORIDE, HCl.

102. **Hydrochloric acid** is a very important compound of chlorine. It may be prepared by the action of sulphuric acid upon common salt (NaCl).

EXPT. 126.—Place several lumps of rock salt (sodium chloride) in the flask which was used for the preparation of chlorine (fig. 54). Then dilute some strong sulphuric acid with an equal volume of water, and pour it into the flask. Hydrochloric acid gas will be evolved as soon as a gentle heat is applied: $NaCl + H_2SO_4 = HCl + NaHSO_4$. The gas is heavier than air. It should be collected by downward displacement, since it dissolves easily in water, producing liquid hydrochloric acid. Fill three jars with the gas, and cover each with a glass plate.

103. Preparation and Properties of Hydrochloric acid gas.

EXPT. 127.—Uncover a jar of this gas, and notice that fumes are given off which turn blue litmus paper red, and are therefore acid in reaction.

Introduce a burning wax taper into a jar of the gas, and notice that the flame is immediately extinguished.

Rinse out a dry cylinder with strong ammonia solution, and place it mouth to mouth with a jar of hydrogen chloride gas (fig. 56, p. 106). Note that opaque white fumes of ammonium chloride are formed in the vessels.

Invert a jar of hydrogen chloride gas, and place its open mouth in water. The water will rise into the jar as the gas dissolves in the liquid. Probably a space will remain in the jar unfilled with water: this is due to the presence of air, which has not been displaced by hydrogen chloride.

104. Volume of Hydrogen present in Hydrogen chloride.—Hydrochloric acid gas may be shown to be composed of equal volumes of hydrogen and chlorine, by electrolysing its solution in water.

The following experiment may be more easily made in order to show that this gas contains half its volume of hydrogen. It depends upon the fact that when sodium is exposed to hydrogen chloride it combines with the chlorine and sets free the hydrogen. Solution of sodium in mercury (sodium amalgam) is most conveniently employed for this purpose.

EXPT. 128.—Clean and dry the tube which was used in Experiment 54 (page 51), and fill it by downward displacement with pure dry hydrochloric acid gas; then close the tube with a rubber cork. Now prepare a little sodium amalgam by placing about 5 c.c. of mercury in a mortar and adding small pieces of sodium one by one. Each piece of sodium should be firmly pressed against the mortar beneath the mercury with the pestle until it combines with that metal with production of heat. Sufficient sodium must be introduced to make the amalgam slightly pasty.

Now measure the volume of the amalgam; then pour it quickly into the tube containing the hydrogen chloride, and at once replace the cork. Shake up the amalgam with the gas for about five minutes, then open the tube under water in a cylinder, bring the water inside and outside the tube to the same level, and mark by means of a rubber ring on the tube the level at which the water stands. If the volume which was occupied by the amalgam is allowed for, it will be found that half the original volume of gas is left. Close the mouth of the tube with the thumb and open the tube close to a flame; the gas will be found to burn (54), and the flame will be probably coloured yellow by the sodium vapour contained in the hydrogen.

This experiment shows that hydrochloric acid gas contains half its volume of hydrogen gas.

THE CHLORIDES.

105. Formation of Chlorides from Hydrogen chloride.—Many metals, when they are placed in contact with hydrochloric acid (HCl), have the power of replacing the hydrogen of the acid. The products in such cases are hydrogen gas and a compound of the metal with chlorine. When this change occurs the acid character of the hydrogen chloride wholly or partially disappears. The metallic compound which is formed is called a chloride. Thus in the preceding experiment hydrogen and sodium chloride were produced by the action of sodium on the acid.

EXPT. 129.—Place about 15 c.c. of dilute hydrochloric acid in a boiling-tube and drop in a few pieces of magnesium ribbon. The magnesium will dissolve with effervescence, and the hydrogen may be kindled as it passes away from the tube. As soon as no more magnesium can be dissolved, evaporate the liquid considerably, and allow it to crystallise. Colourless crystals of magnesium chloride ($MgCl_2.6H_2O$) will be deposited.

It is not necessary to introduce the metal in the free condition into the acid in order to produce a chloride. A suitable compound of the metal may be added, such as the oxide, the hydroxide, or the carbonate.

EXPT. 130.—Add powdered barium carbonate gradually to a small quantity of hydrochloric in a small porcelain-dish. Continue the addition until the last portion of the carbonate causes no effervescence and does not dissolve:—

$$BaCO_3 + 2HCl = BaCl_2 + H_2O + CO_2.$$

Evaporate this solution until it is reduced to about one-half its volume, and allow it to cool. Crystals of barium chloride ($BaCl_2.2H_2O$) will form in the liquid.

106. Preparation of Chlorides by Double Decomposition, or Metathesis.—A chloride which is insoluble, or only slightly soluble, in water may be conveniently prepared by the method of double decomposition. It consists in adding solution of a chloride to the solution of a salt of the metal whose chloride is to be prepared. The chloride will be precipitated from the mixed solutions.

Thus if sodium chloride solution is added to solution of lead nitrate a white precipitate of lead chloride is produced and sodium nitrate remains in solution ($2NaCl + PbN_2O_6 = PbCl_2 + 2NaNO_3$).

Expt. 131.—Add sodium chloride solution, or dilute hydrochloric acid, to some lead nitrate solution contained in a test-tube. A white precipitate will form. Add more water and heat to boiling; the white precipitate will dissolve if sufficient water is added, since it is soluble in boiling water. Now allow the solution to cool. Characteristic slender crystals of pure lead chloride will form. These may be collected by filtering or decanting the liquid.

107. Tests for Hydrochloric acid and Chlorides.—When hydrochloric acid is heated with manganese dioxide, chlorine gas is evolved, and may be recognised by its power of rapidly bleaching moistened litmus paper. When a chloride is finely powdered and intimately mixed with powdered manganese dioxide, and is then heated with sulphuric acid, chlorine is also evolved.

The solution of a soluble chloride gives a white precipitate when it is mixed with silver nitrate solution; the precipitate darkens in colour on exposure to bright daylight, and is insoluble in nitric acid.

Acids and Salts.

108. Nature of Acids and of Salts.—The above examples give some insight into the general characteristics of acids and of salts.

Acids invariably contain hydrogen, as hydrochloric acid has been proved to do. They are also usually sour in taste, and capable of turning blue litmus red.

The hydrogen contained in an acid may be wholly or partially replaced by a metal, with production of a salt. This was seen in the case of the formation of magnesium chloride by the action of magnesium on hydrochloric acid.

Salts may commonly be formed, as in the special cases already mentioned, by the action of a metal, its oxide, its hydroxide, or its carbonate upon an acid.

Other methods of forming salts have been described, such as the process of double decomposition (106), the direct combination of elements (33), and the replacement of one metal from its salt by another metal (32, Expt. 32).

Further information on these matters will be found in paragraph 144.

SECTION VIII.

AMMONIA.
NITROUS OXIDE, NITRIC OXIDE.
NITRIC ACID, NITRATES.

AMMONIA, NH_3.

109. Ammonia produced from Organic Matter.—Ammonia is given off when organic matter containing nitrogen decays or is heated strongly. At one time the supply of ammonia was entirely derived from animal refuse by heating it out of contact with air. It is now almost entirely prepared from the ammoniacal liquor obtained as a by-product in the manufacture of coal-gas.

EXPT. 132.—Heat a little dried albumen or gelatin in a test-tube. The peculiar pungent smell of ammonia will be perceived, although it is more or less masked by a less agreeable odour. Hold a strip of moistened red litmus paper in the upper part of the test-tube; the litmus will become blue owing to the alkaline action of the ammonia.

EXPT. 133.—Ammonia may be obtained from gelatin more readily and in a purer condition if the gelatin has been previously intimately mixed with powdered soda-lime. Introduce this mixture into a test-tube, and cover it with a layer of the powdered soda-lime. Heat the soda-lime to redness, and then strongly heat the mixture. Ammonia will be given off unaccompanied with the other substances which produce a disagreeable smell.

110. Ammonia produced from Ammonium chloride or Sulphate.—Ammonium chloride is made on a large scale by neutralising the ammoniacal liquor of the gasworks by hydrochloric acid; and ammonium sulphate is made in a similar way by substituting sulphuric acid for hydrochloric acid.

If either the chloride or the sulphate of ammonium is mixed with lime and gently heated, ammonia gas is evolved.

Expt. 134.—Powder a little ammonium chloride (*sal ammoniac*) in a mortar, and mix it with about an equal quantity of slaked lime in fine powder: the ammonia gas escaping from the mixture will be readily smelt; the gas may be expelled more rapidly and completely by gently heating the mixture:

$$2NH_4Cl + CaH_2O_2 = 2NH_3 + CaCl_2 + 2H_2O.$$

III. Ammonia prepared from strong Ammonia solution.—Ammonia gas may be conveniently obtained by expelling it from its solution in water by means of heat.

Expt. 135.—Pour a little strong ammonia solution (0·88 sp. gr.) into a small flask, and close the flask with a cork into which a straight piece of glass tube eight or nine inches long has been fitted (fig. 55). When the flask is gently heated, the liquid will at once appear to boil, owing to the evolution of bubbles of ammonia gas.

Since this gas is much lighter than air and is very soluble in water, it should be collected by direct displacement of the air from a *perfectly dry* jar, as has been already described for hydrogen (Expt. 61, p. 57). The jar is known to be filled with ammonia when a strip of moistened turmeric paper held in its mouth immediately becomes brown.

Ammonia gas does not burn in cold air, but it burns readily in strongly heated oxygen gas or in air.

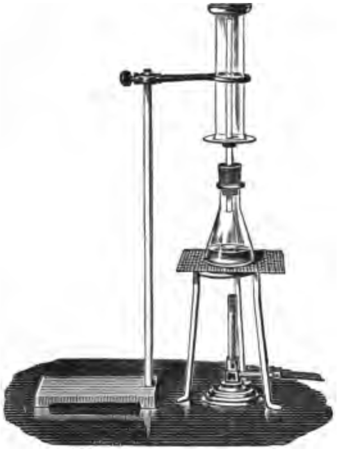
Expt. 136.—Show this by holding a Bunsen-flame under the end of the delivery-tube from which the ammonia is escaping. A pale yellowish-green flame of burning ammonia will be seen.

If the ammonia is kindled as the end of the tube is passed into a jar of oxygen, the ammonia will also continue to burn in that gas.

Ammonia is extremely soluble in water, and must therefore be collected either by displacement or over mercury.

Expt. 137.—Fill a jar with ammonia and place it mouth downwards in a vessel of water. The liquid will rapidly absorb the gas and will rise in the jar until it fills the space formerly occupied by the ammonia. A small space, however, will frequently remain filled with the air which was present in the ammonia.

Fig. 55.



Preparation of Ammonia gas from its Solution.

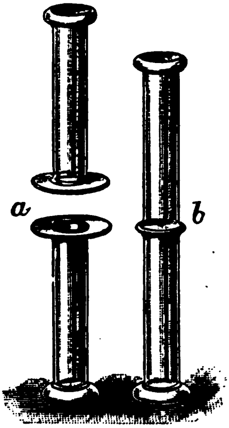
Expt. 138.—Pour a little water rapidly from a small beaker into a jar of ammonia gas, and at once cover the jar with a glass plate or with the hand. Then shake the water in the jar, and pour it out. The liquid will be found to possess the pungent smell and the action upon litmus and turmeric papers which characterise ammonia gas.

This liquid is in fact weak solution of ammonia. The solution is prepared commercially in large quantities by passing ammonia gas for some time into cold water. It gives off a portion of its gas slowly at ordinary temperatures, and evolves the gas rapidly when it is heated, as has been already proved (Expt. 135).

EXPT. 139.—Pour a little strong hydrochloric acid into a glass jar, and close the mouth of the jar with a glass plate. Shake the acid about, and allow the liquid to run out by slipping aside the glass plate for a moment.

Now place this jar in an inverted position over another jar containing ammonia gas (fig. 56 *a*), and withdraw the glass plates, so that the mouths of the jars are in contact (*b*). The hydrochloric acid gas and the ammonia gas in the two jars will now freely intermingle. Dense opaque white fumes of solid ammonium chloride will immediately be formed: $NH_3 + HCl = NH_4Cl$.

FIG. 56.



COMBINATION OF HYDROGEN CHLORIDE WITH AMMONIA.

These fumes may also be obtained by dipping a glass rod into strong hydrochloric acid, and holding it either in the ammonia gas as it issues from the delivery-tube of the apparatus (fig. 55), or in a jar which has been previously filled with the gas.

112. **Tests for Ammonia gas.**—Ammonia gas may be readily recognised by its pungent smell; by turning moistened red litmus paper blue and moistened turmeric paper reddish-brown; also by giving opaque white fumes when it is brought into contact with hydrochloric acid gas, or near a surface moistened with the *strong* hydrochloric acid solution.

OXIDES OF NITROGEN.

113. **Five Oxides of nitrogen** are known. Only two of them will be described here: nitrogen monoxide or nitrous oxide, N_2O, and nitric oxide, NO.

NITROGEN MONOXIDE, OR NITROUS OXIDE GAS, N_2O.

114. **Preparation of Nitrogen monoxide.**—When solid ammonium nitrate is heated, it melts; and when the liquid is further heated, it decomposes into steam and nitrogen monoxide:

$$NH_4NO_3 = N_2O + 2H_2O.$$

EXPT. 140.—Cleanse the flask which was used in the preparation of chlorine (fig. 54, p. 97). Close it with a singly perforated cork, bearing a delivery-tube which has been bent twice at right angles. Replace the washing-bottle by a flask which is closed by a doubly perforated cork, bearing tubes which pass just through the cork. Weight the flask by pouring shot into it, so that it may be sunk into a pan of cold water. This flask serves to condense the steam which escapes with the gas, and to cool the gas. Now heat the ammonium nitrate, and collect the nitrogen monoxide by displacement, since it is much heavier than air, and is somewhat soluble in water.

EXPT. 141.—Insert a splinter of wood with a glowing end into a jar of the gas. The splinter will be kindled into flame, but a greenish halo will play around the flame, which is not seen when the flame is burning in oxygen.

Show further that nitrous oxide gives no red colour when it is mixed with nitric oxide as oxygen does. The nitrous oxide which is used for this experiment should be collected over water, since air will probably be present in the gas after its collection by displacement.

115. **Tests for Nitrous oxide.**—The gas has a sweet taste; it is detected by its property of kindling a spark on a wooden splinter into flame, and is distinguished from oxygen by the greenish halo around the flame; it also differs by not colouring nitric oxide.

NITRIC OXIDE GAS, NO.

116. **Preparation of Nitric oxide from Nitric acid.**—This gas may be prepared by the action of diluted nitric acid on copper: $3Cu + 8HNO_3 = 2NO + 3Cu(NO_3)_2 + 4H_2O$.

EXPT. 142.—Cleanse the apparatus which has been already used for the preparation of hydrogen (fig. 34, p. 56); place some copper clippings or turnings in it, and pour in water until the end of the funnel-tube is covered. Then add strong commercial nitric acid gradually until a reddish-brown gas appears inside the bottle. Allow this gas to bubble off through water. After a short time the colour of the gas in the bottle will become much less intense, and if it is collected over water, in the same way as hydrogen gas was collected, it will become colourless.

Expt. 143.—Invert a jar filled with water in water, and fill it with nitric oxide gas. Then either pass oxygen up into it, or remove the jar from the water and allow it to stand with its mouth open to the air. The entrance of the oxygen, or of the air, into the nitric oxide will at once produce a reddish-brown colour.

The chief origin of the coloured gas which was seen in the preparation-bottle (Expt. 142) is now explained. The bottle was at first filled with air, and as soon as the first portions of the nitric oxide gas were produced, they mingled with the free oxygen of the air, and formed nitrogen peroxide. Some nitrogen peroxide is, however, also produced directly from the nitric acid. The coloured gas is rapidly dissolved by water.

117. **Test for Nitric oxide.**—Nitric oxide is colourless: but when nitric oxide is mixed with free oxygen, it is converted into reddish-brown nitrogen peroxide.

PREPARATION OF NITRIC ACID (HNO_3) BY DISTILLATION.

118. **When Potassium nitrate** is mixed with strong sulphuric acid and the mixture is heated, nitric acid is formed: $KNO_3 + H_2SO_4 = HNO_3 + KHSO_4$.

The nitric acid may be separated from the solid potassium sulphate, which is produced at the same time, and from excess of sulphuric acid, by the process of distillation.

Expt. 144.—Arrange again the apparatus which was used for the distillation of water (fig. 39, p. 62). Introduce some solid nitre or potassium nitrate into the retort through the tubulure. Then place a funnel in the tubulure, pour in about double the measure of strong sulphuric acid, and proceed to distil over the nitric acid. A yellow liquid will trickle down the neck of the retort into the flask. This liquid is somewhat impure nitric acid.

The liquid which remains in the retort after the distillation should be poured out as soon as it is cold; the retort may then be rinsed with water.

119. Most metals are converted into their nitrates when they are acted on by nitric acid, but some metals are simply converted into their oxides.

Expt. 145.—Pour a little of the strong nitric acid from the condensing-flask (Expt. 144) upon some small pieces of copper in a test-tube. A reddish-brown gas will escape in large quantity, and a blue solution containing copper nitrate will be produced. The salt may be obtained in blue crystal by concentrating its solution sufficiently by evaporation.

Expt. 146.—Pour another portion of the strong nitric acid upon a small piece of tin. A red gas will be evolved as before, and the tin will be converted into white tin dioxide, but will not be dissolved.

120. Test for Nitric acid.—Strong nitric acid emits pungent fumes, and evolves reddish-brown gas when it is brought into contact with copper.

The Nitrates.

121. Most solid nitrates, when they are heated, give off either oxygen alone, or a mixture of oxygen with a reddish-brown gas known as nitric peroxide.

Expt. 147.—Heat a little solid potassium nitrate in a hard glass test-tube; the salt will melt, and will slowly give off bubbles of gas when it is more strongly heated. A glowing splinter of wood will be kindled into flame by this gas, proving that the gas is oxygen.

Expt. 148.—Heat a little solid lead nitrate in a test-tube; reddish-brown gas will be evolved, consisting of a mixture of oxygen with nitric peroxide. The presence of the nitrogen peroxide is proved by the colour of the gas, and the presence of oxygen may be shown by introducing a glowing splinter of wood.

122. Test for Nitrate.—If fairly strong sulphuric acid is added to a nitrate, and a few copper turnings are dropped into the liquid, a reddish-brown gas is given off. The production of this gas is hastened by shaking the tube and by heating the mixture.

SECTION IX.

SULPHUR, HYDROGEN SULPHIDE, SULPHIDES.
SULPHUR DIOXIDE, SULPHUROUS ACID, SULPHITES.
SULPHUR TRIOXIDE, SULPHURIC ACID, SULPHATES.

SULPHUR.

Sulphur is found in nature in large quantities in volcanic regions; it is also produced from various commercial by-products which contain it.

123. Allotropic Forms of Sulphur.—Sulphur occurs in several *allotropic* modifications, of which the following may be mentioned:

(*a*) Octahedral crystals.
(*b*) Prismatic or needle-shaped crystals.
(*c*) Plastic and elastic form.
(*d*) White, or amorphous form.

The following experiments may be performed to illustrate the preparation of these different varieties of sulphur. Experiments 149, 150, and 151 require care, and should be performed under supervision.

EXPT. 149.—Shake up a little flour of sulphur for some minutes with a few c.c. of carbon disulphide; take care to keep the liquid away from a flame, since it is very volatile and the vapour is inflammable. Filter the liquid through a dry filter into a small dry beaker; cover the beaker with filter-paper and place it in a draught-closet or in the open air, so as to allow the carbon disulphide to evaporate slowly.

When all the liquid has disappeared, yellow octahedral crystals of sulphur will be seen in the beaker.

Expt. 150.—Half fill a small porcelain-dish with lumps of stick sulphur, and carefully heat the dish with a small flame until the sulphur is entirely melted. Allow the liquid to cool until a thin crust of solid sulphur has formed upon the surface. Then make two holes in this crust with a stout wire or round file at opposite sides of the dish. Now grasp the dish with the crucible tongs, and pour out the still liquid sulphur through one of the holes. As soon as the dish is sufficiently cool to be handled, remove the crust. A mass of transparent, honey-coloured, slender, tough crystals of sulphur will be seen beneath the crust and lining the dish.

Put the dish aside for a time, and note that the appearance of the crystals alters. They will become opaque and bright yellow, and will at the same time become extremely brittle.

Expt. 151.—Heat a few pieces of sulphur in a test-tube very gently, constantly noting the colour, appearance, and consistency of the sulphur as its temperature is gradually raised. The colour will at first be pale and the liquid will be perfectly limpid; but the colour will gradually darken to brown and the liquid will become less limpid, until at last it is so thick that the tube may be inverted without causing the sulphur to flow out. When the sulphur is heated more strongly, however, it will become limpid once more and almost black in colour. As the sulphur cools, these changes will occur again, but in the reverse order.

While the dark-coloured liquid is limpid at the highest temperature pour it out into cold water. The sulphur obtained in this way by quickly cooling the liquid will be found to be 'plastic,' soft, and elastic. But this character will be gradually lost if it is put on one side for a day or two.

Expt. 152.—Add dilute hydrochloric acid gradually to a little yellow ammonium sulphide solution. Sulphur will be precipitated in the form of a fine white powder. This is known as 'milk of sulphur.'

NOTE.—Any of the above substances may be shown to consist of sulphur by kindling them, when they will burn with the characteristic blue flame of sulphur, and will produce a pungent-smelling gas called sulphur dioxide.

HYDROGEN SULPHIDE GAS, H_2S.

124.—This gas is often called sulphuretted hydrogen. It is readily prepared in a manner similar to carbon dioxide (Expt. 94, p. 80) by the action of diluted hydrochloric or sulphuric acid upon ferrous sulphide, $FeS + 2HCl = H_2S + FeCl_2$. It should be collected by downward displacement, since it is somewhat soluble in water. The gas is very poisonous, and has a foul smell, and must therefore be prepared in the draught-closet. It burns explosively when it is mixed with air and kindled. The student may collect the gas from the general laboratory-apparatus, if it is available.

125. Properties of Hydrogen sulphide.—Moisten a piece of filter-paper with lead acetate solution, and expose the paper to the gas; it will become dark brown.

Burn the gas in a glass cylinder and note the deposition of yellow sulphur on the inside of the glass. Close the cylinder at once with a glass plate, and shake some water which has been faintly coloured with potassium dichromate solution round inside it. The colour of the liquid will change to green, showing that sulphur dioxide has been formed by the combustion.

THE SULPHIDES.

126. When sulphur is heated with a metal, the two elements usually combine and form a sulphide.

EXPT. 153.—Introduce a small piece of sulphur into a test-tube, and place above it a piece of tinfoil rolled into a ball. Heat the sulphur until its vapour comes into contact with the tinfoil, and notice the formation of tin sulphide.

127. A convenient method of forming sulphides consists in treating a solution of a metallic salt with hydrogen sulphide gas. Hydrogen sulphide is largely used in the process of chemical analysis for the purpose of precipitating certain metals from their solutions as insoluble sulphides. The darkening of the lead acetate solution on paper, which has been already referred to (125), is due to the formation of dark brown lead sulphide in this way.

EXPT. 154.—Pass a stream of hydrogen sulphide gas in bubbles through a solution of copper sulphate, of antimony chloride, and of arsenious oxide, and notice the appearance of the precipitate which is produced in each case.

SULPHUR DIOXIDE, OR SULPHUROUS OXIDE GAS, SO_2.

128. Preparation of Sulphur dioxide from Sulphuric acid.—This gas is readily made by burning sulphur in oxygen (Expt. 50, p. 47). It may also be prepared from strong sulphuric acid by heating the acid with copper.

EXPT. 155.—Cleanse the preparation flask (fig. 54, p. 97), and place some copper turnings in it. Then pour in strong commercial sulphuric acid, and heat the mixture. Sulphurous oxide will be evolved when the acid has been heated nearly to boiling:

$$Cu + 2H_2SO_4 = SO_2 + CuSO_4 + 2H_2O.$$

The gas may be cooled and dried by passing it through strong sulphuric acid. It is then collected by displacement, since it is much heavier than air and is extremely soluble in water.

129. Properties of Sulphur dioxide.—This gas possesses an extremely pungent smell, and is identified by the following properties, of which the first is the most characteristic.

EXPT. 156.—Pour some dilute solution of potassium dichromate into a jar of the gas, and shake the liquid round in the jar; also hang paper moistened with the dichromate solution in the jar; the reddish colour of the dichromate will be rapidly changed to bluish-green. Prove also that the gas is freely soluble in water, and that its solution is acid since it turns blue litmus paper red.

SULPHUROUS ACID (H_2SO_3) AND SULPHITES.

130. It has been proved (Expt. 156) that sulphur dioxide gas is soluble in water. The solution may be inferred to be a solution of sulphurous acid, since it has an acid reaction to test-paper, and is capable of decomposing carbonates. The sulphurous acid in this solution is gradually changed into sulphuric acid by exposure to air.

Expt. 157.—Add to some *freshly-made* sulphurous acid solution a little dilute hydrochloric acid and then a few drops of barium chloride solution; either no precipitate will be produced or only a slight turbidity will appear.

Now add to some *old* sulphurous acid solution the same two reagents; a copious white precipitate will form, showing that the sulphurous acid has been changed by oxidation into sulphuric acid (135).

The salts termed sulphites, which are formed by neutralising sulphurous acid with an oxide or a hydrate, are more stable than the acid.

Expt. 158.—Place some sodium sulphite in a test-tube; pour upon it a little dilute hydrochloric acid, and warm the mixture. Sulphur dioxide gas will be evolved, and may be detected by its smell and by its power of changing the colour of paper which has been dipped into potassium dichromate solution from yellow to green.

SULPHUR TRIOXIDE, SO_3.

131. **Sulphur trioxide** is not readily prepared on a small scale. When sulphur is burnt in air, only a very small quantity of the sulphur is converted into sulphur trioxide, the main product being sulphur dioxide. The latter gas may be made to unite with oxygen by the influence of certain substances at a high temperature. Finely divided platinum may be used for this purpose; it does not undergo any permanent change during its use.

Expt. 159.—Prepare some platinised asbestos by dipping 'woolly' asbestos into platinum chloride solution and then heating it in the Bunsen-burner. Repeat this operation several times until the asbestos assumes a decidedly grey colour from the separation of finely divided platinum upon its surface.

Place a little of this platinised asbestos in a piece of combustion tube. Connect one end of the tube with an aspirator (fig. 38, p. 60) or with a filter-pump, and the other with a washing-bottle containing sulphurous acid solution. The wash-bottle must be so arranged that when the aspirator is set going it will draw a stream of air through the acid.

Now heat the asbestos to bright redness, and draw a stream of air through the apparatus. As the air passes through the sulphurous acid solution it will remove a little sulphur dioxide, so that a mixture of air with sulphur dioxide gas will pass over the platinised asbestos. White fumes of sulphur trioxide will be seen. These fumes produce sulphuric acid when they are drawn through water as is proved by the water reddening blue litmus paper, and yielding a precipitate with barium chloride (135).

SULPHURIC ACID, H_2SO_4.

132. Preparation of Sulphuric acid.—This acid is manufactured in very large quantities by the action of oxygen upon sulphur dioxide in the presence of water. Under ordinary conditions the action is very slow. But if the oxygen is presented to the sulphur dioxide by means of a suitable oxide the oxidation is much more rapid. On a large scale the oxygen of the higher oxides of nitrogen is used for the purpose.

The process may be imitated on a small scale by oxidising a solution of sulphur dioxide by means of nitric acid.

EXPT. 160.—Prepare a fresh solution of sulphur dioxide by passing the gas into some distilled water, which has been recently boiled in order to expel dissolved oxygen.

Test this solution for sulphate by means of hydrochloric acid and barium chloride (135); probably no precipitate will appear.

Now add a few c.c. of strong nitric acid to a little of the sulphur dioxide solution in a four-ounce flask, and boil the liquid for some time. Test this solution with hydrochloric acid and barium chloride solution. A copious precipitate of barium sulphate will form, showing that the sulphur dioxide solution has been oxidised to sulphuric acid by the nitric acid.

133. Properties of Sulphuric acid.—A few experiments are given below to illustrate the properties of sulphuric acid. When the acid is strong it is very corrosive; it rapidly destroys the clothes and causes painful burns when it is in contact with the skin (see *Caution*, p. 26).

EXPT. 161.—Dissolve a few lumps of sugar in as little water as possible in a small beaker. Place the beaker on a dinner-plate, and pour into the liquid about its own volume of strong sulphuric acid. A violent action will take place, attended with escape of sulphur dioxide, and production of a black mass of charcoal. The strong acid chars most organic substances in this way by removing from them the elements of water.

Expt. 162.—Place a drop of dilute sulphuric acid upon a filter-paper, and gently heat it over the Bunsen burner. As the acid becomes concentrated by evaporation the paper will be charred.

Expt. 163.—Pour a thin stream of the strong acid into a little water in a beaker. Notice that the beaker becomes hot, owing to the combination of the acid with the water.

Never pour water into strong sulphuric acid, since this might cause a serious accident, owing to the rapid production of intense heat.

Expt. 164.—Pour a little strong sulphuric acid into a beaker; mark the level of the upper surface of the acid by means of a gummed strip of paper on the side of the beaker, and note down the weight of the beaker and acid. Then allow the open beaker to remain in a safe place for a week, and note the increase of volume and of weight which has occurred in the acid. This increase in volume and weight is caused by the acid absorbing moisture from the air. The strong acid is therefore a powerful 'desiccator,' and it is often used for absorbing the moisture from gases or drying them.

THE SULPHATES.

134. The sulphates are a very important class of salts. They may be prepared by the general methods given in paragraph 108.

Expt. 165.—Prepare zinc sulphate crystals ($ZnSO_4.7H_2O$) by allowing zinc to act upon dilute sulphuric acid in a small beaker until no more zinc can be dissolved, then pouring off the liquid from the undissolved zinc, concentrating it by evaporation, and allowing it to crystallise.

Expt. 166.—Add potassium hydroxide solution gradually to some dilute sulphuric acid until the liquid is just alkaline and turns red litmus paper blue; concentrate the liquid by evaporation, and allow it to crystallise. Potassium sulphate crystals (K_2SO_4) will be obtained.

Expt. 167.—Prepare insoluble barium sulphate by adding barium chloride solution to dilute sulphuric acid; filter, and prove that the precipitate does not dissolve in the common dilute acids.

135. **Test for Sulphates.**—The solution of a sulphate which has been acidified with hydrochloric acid gives a white precipitate when it is mixed with barium chloride solution; the precipitate is insoluble in hydrochloric acid, and in dilute acids generally.

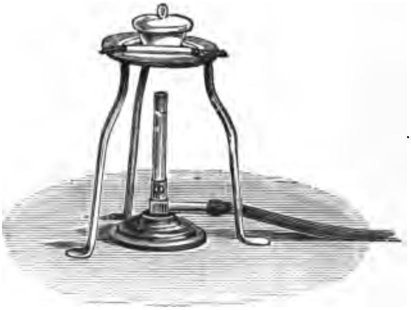
SECTION X.

CONSTANCY OF COMPOSITION OF COMPOUNDS.
EQUIVALENT WEIGHTS.
ACIDS, BASIC SUBSTANCES AND SALTS.
ACIDIMETRY AND ALKALIMETRY.
MISCELLANEOUS EXAMPLES.

CONSTANCY OF COMPOSITION OF COMPOUNDS.

A Chemical Compound invariably contains the same constituents combined in the same proportion by weight. This is known as the Law of Definite Proportions. Several experiments have already been described which illustrate this law. A few others are appended below.

FIG. 57.



COMBUSTION OF MAGNESIUM.

136. Combination of Magnesium with Oxygen.—Magnesium may be readily converted into magnesium oxide by burning it in the air. The study of this change by means of the balance will afford another proof that the relation by weight of the constituents of a compound substance is constant.

Expt. 168.—Carefully weigh a porcelain crucible about 1·75 inches in diameter, and note down the weight. Clean some magnesium ribbon by rubbing it with sandpaper, and weigh out 0·4 gram of the metal. Cut the metal into small pieces, and drop it into the crucible.

Place the crucible upon a pipeclay triangle and tripod (fig. 57), and nearly cover it with the lid, leaving a small open space; then gradually heat it until the bottom is red-hot. As soon as the metal ceases to glow, take off the lid and heat the grey mass in the crucible over the blowpipe-flame until it becomes white. Now weigh the cool crucible and its contents again on the balance, and deduct from this weight the weight of the empty crucible. The difference will give the weight of the magnesium oxide which has been formed.

Calculate the ratio of the weight of the original magnesium to that of the magnesium oxide (MgO) formed. This ratio will remain unchanged if the experiment is repeated.

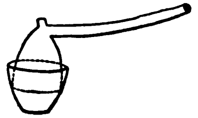
137. Combination of Copper with Sulphur.

137. Combination of Copper with Sulphur.—When copper is heated with sulphur, it is converted into sulphide, but the conversion is incomplete unless the air is excluded.

Expt. 169.—Weigh a crucible on the balance, and then weigh into it one gram of bright copper turnings or filings. Cover the metal completely with powdered sulphur, and support the crucible on a triangle or tripod (fig. 57).

Now fit the bowl of an inverted clay tobacco-pipe into the crucible (fig. 58), and conduct a gentle stream of coal-gas through the pipe-stem by means of a rubber tube. By this device the contents of the crucible may be gently heated in an atmosphere of coal-gas.

Fig. 58.



Formation of Copper Sulphide.

As soon as the blue flame of the burning sulphur has disappeared, allow the crucible to cool in the current of coal-gas and weigh it. Now sprinkle a little more sulphur on the contents of the crucible, repeat the heating and cooling in coal-gas, and weigh again. Continue these operations until the crucible and its contents cease to increase in weight.

Calculate the ratio of the weight of the copper to that of the cuprous sulphide (Cu_2S) formed.

EQUIVALENT WEIGHTS OF ELEMENTS.

138. The Equivalent Weight of an element is the weight which will enter into combination with, or displace from combination, one part by weight of hydrogen.

Most elements possess one equivalent only, but certain elements possess two or more equivalents. Thus tin forms two series of compounds which are distinguished as stannous and stannic compounds. The equivalent of tin in stannous compounds is 59, and in stannic compounds 29·5.

Since some elements do not combine with hydrogen to form definite compounds, and since hydrogen is so extremely light that it is difficult to weigh it with accuracy, indirect methods of determining equivalents are commonly used.

The error incurred in weighing hydrogen can sometimes be obviated by replacing the hydrogen of an acid by the element. The *volume* of hydrogen evolved can then be ascertained, and its weight may be calculated from its volume.

In other cases the weight of the element is determined which combines with a fixed weight of some other element, the equivalent of which is known. Thus the equivalent of oxygen is known to be 8, and one gram of Mg combines with 0·67 gram of oxygen (136); hence the equivalent of magnesium, compared with oxygen as unit, is $\frac{1}{0{\cdot}67} = 1{\cdot}5$: accordingly, compared with hydrogen as unit, the equivalent will be $1{\cdot}5 \times 8 = 12$.

Many of the experiments which have been already performed will serve to determine the equivalents of elements, when the equivalent of one of them, compared with hydrogen as unit, is known.

It will usually be sufficient, in the following examples, to give the relations of the weights of the elements to each other. These numbers may easily be converted into hydrogen equivalents by reference to the Table of Equivalents given in the Appendix.

Three methods of determining equivalents are described below:—(1) direct replacement of one metal by another; (2) conversion of the metal into oxide; and (3) measurement of the volume of hydrogen displaced by a metal during its solution by an acid.

EQUIVALENTS BY REPLACEMENT OF METAL BY METAL.

139. **The Replacement of a Metal from the Solution of its Salt by another Metal** was the earliest means adopted for ascertaining equivalent weights. When a suitable metal is placed in the solution of a salt of another metal, it will dissolve and will liberate an equivalent weight of the other metal contained in the salt.

Thus when zinc or iron is placed in a cold solution of copper sulphate, the metal will be dissolved, and a definite weight of copper will be separated. The weight of copper thus separated will always bear a certain fixed relation to the weight of the metal which has been dissolved. The *equivalent* weights of the metals, which have been thus obtained, may be used for calculating the hydrogen equivalents, as has been explained above. In this example the hydrogen equivalent of copper must be obtained in order to calculate the hydrogen equivalent of the other metals.

EXPT. 170.—**Replacement of Copper by means of Zinc.**—Weigh out one gram of *pure* zinc foil and place it in a weighed porcelain-dish in somewhat dilute cold solution of copper sulphate: a dark-coloured coating of metallic copper will immediately be deposited upon the zinc.

If the copper sulphate is present in sufficient quantity, the whole of the zinc will ultimately be dissolved. The completion of the process may be hastened by gently warming the liquid. When the zinc has been dissolved, the liquid must be distinctly blue in colour, proving that copper sulphate is present in excess.

As soon as no hard pieces of zinc can be felt or seen when the deposit is stirred with a glass rod, pour off the liquid carefully, without removing any of the copper, and wash the metallic powder carefully, first with water and finally with alcohol. Then dry it in the steam oven, and quickly weigh the dish and its contents as soon as they are cold.

Calculate the hydrogen equivalent from the weight of copper deposited.

140. **Other Replacements of Metals**, which may be carried out in a similar manner to that already described, are given below.

Expt. 171.—Estimate the weight of copper which is precipitated by dissolving one gram of clean iron turnings in excess of copper sulphate solution.

Expt. 172.—Estimate the weight of silver which is precipitated from silver nitrate solution by 0·5 gram of copper, taking care that excess of silver solution is employed, and that no silver is lost.

Expt. 173.—Estimate the weight of lead which is precipitated by dissolving 0·5 gram of zinc in excess of lead acetate solution.

Calculate the H equivalents of Cu, Ag and Pb from the results obtained.

The Results of the Experiments should be as follows :—

1	gram of	zinc	precipitates	0·97	gram of	copper
1	„	iron	„	1·13	grams of	copper
1	„	copper	„	3·40	„	silver
1	„	zinc	„	3·20	„	lead

From the above results it is possible to calculate the weights of the metals which are equivalent to each other.

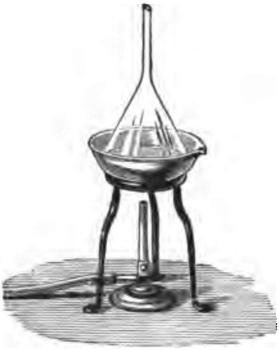
Thus 1 gram of Fe is equivalent to 1·13 grams of copper, also 1 gram of copper is equivalent to 3·40 grams of silver. Hence it is inferred that 1 gram of Fe is equivalent to 1·13 × 3·40 = 3·84 of silver. If the hydrogen equivalent of Fe is 28, then the hydrogen equivalent of silver will be 3·84 × 28 = 107·5.

EQUIVALENTS BY CONVERSION INTO OXIDES.

141. Most metals are converted by nitric acid either into nitrates or into oxides. The oxide is produced in most cases if the excess of acid is evaporated completely after the action on the metal is complete, and the residue is then strongly heated. This process of converting metals into their oxides furnishes a simple means of obtaining the oxygen equivalents of the metals.

EXPT. 174.—**Estimation of the Equivalent of Copper by converting it first into Nitrate and then into Oxide.**—Weigh out one gram of copper turnings into a small porcelain evaporating dish, which has been previously weighed, and add a few c.c. of strong nitric acid. Cover the dish immediately with a funnel in order to prevent loss by spirting. As soon as the action has become moderate, place the dish on a pipeclay triangle, supported on a tripod-stand (fig. 59), and heated very gently with a small flame.

FIG. 59.



CONVERSION OF COPPER INTO COPPER OXIDE.

When no more effervescence occurs, remove the funnel, rinsing it into the dish with the wash-bottle, and evaporate the blue solution to dryness on the water-bath (fig. 40, p. 68). Then heat the blue copper nitrate, gradually increasing the temperature until no red fumes are given off and the residue is black.

Allow the dish to cool and weigh it. Then heat it again, and once more cool and weigh it. Repeat these operations until the weight is constant. The increase of weight which the copper has undergone will be the weight of oxygen which one gram of copper combines with to form its oxide.

Thus, supposing that 1 gram of a metal increases in weight by 0·43 gram. Since the hydrogen equivalent of oxygen is 8, 0·43 gram of oxygen will correspond to $\frac{0 \cdot 43}{8} = 0 \cdot 054$ gram of hydrogen. Hence the equivalent of the metal would be $\frac{1}{0 \cdot 054} = 18 \cdot 52$.

Expt. 175.—Convert tin into tin dioxide by treatment with nitric acid, and ascertain its hydrogen equivalent.

EQUIVALENTS BY VOLUME OF HYDROGEN DISPLACED.

142. As has been already stated (138), the volume of hydrogen displaced from an acid by a metal may be measured in order to obtain its equivalent. In the following experiments it is shown that a given weight of a metal gives off the same volume of hydrogen gas from different acids, and that equal weights of different metals give off different volumes of hydrogen from an acid. The method of calculating the hydrogen equivalent from the volume of hydrogen evolved is also explained.

Expt. 176.—**Estimation of the Volume of Hydrogen evolved from different Acids by the action of Zinc.**

Place in the flask of the apparatus, which has already been described in Expt. 65 (p. 60), one gram of pure distilled zinc, and add 15 c.c. of water and a drop of platinum chloride solution. Half fill the small test-tube with strong sulphuric acid, see that the joints of the apparatus are sound, and proceed with the estimation as was described on page 60. When the flask is cold measure the volume of hydrogen which has been evolved, by reading the volume of water displaced into the cylinder.

In a second experiment weigh out one gram of zinc, introduce into the flask 14 c.c. of water, and fill the small test-tube with strong hydrochloric acid.

Compare the volumes of gas evolved after correcting them for temperature (*Note*, p. 50).

The volumes of hydrogen evolved should be the same in both cases.

EXPT. 177.—**Estimation of the Volume of Hydrogen which is evolved from an Acid by a known weight of Zinc, Magnesium, or Aluminium.**

Estimate in the manner described in the preceding paragraph the volume of hydrogen which is evolved by treating the following weights of the above metals with hydrochloric acid, adding a drop or two of platinum chloride if necessary to start the reaction :—Zinc, 1 gram (possibly already done); magnesium, 0·5 gram; aluminium, 0·2 gram.

Correct the volume (*Note*, p. 50), and calculate from the results which are thus obtained, the weight of each metal which would be required to evolve the *same* volume of hydrogen. These weights will be found to be in the relation of the hydrogen equivalents of these metals, which are given in the Table of Equivalents in the Appendix.

This relation follows from the fact that equal volumes of hydrogen have equal weights, and therefore the weights of different metals which replace equal volumes of hydrogen will be in the same ratio as the equivalents of these metals.

143. **Calculation of the Hydrogen Equivalent.**—In order to calculate the hydrogen equivalent of zinc from the volume of hydrogen gas which it evolves from an acid, it is necessary first to reduce the measured volume of the gas to that which it would occupy at 0° C. (*Note*, p. 50). The weight of that volume is then calculated on the assumption that 1000 c.c. of hydrogen at 0° weigh 0·09 gram.

The change of volume due to variation from standard pressure and to the presence of water vapour are not taken into account, since the errors of experiment are greater than the changes of volume due to these causes.

In a particular estimation, 1 gram of zinc evolved 360 c.c. of hydrogen at 17° C. (*t*).

The volume of this gas at 0° C. $= \frac{V \times 273}{237 + t} = \frac{360 \times 273}{290} = 339$ c.c., and its weight $= \frac{339 \times 0 \cdot 09}{1000} = 0 \cdot 0305$ gram. Accordingly the hydrogen equivalent of zinc $= \frac{1}{0 \cdot 0305} = 32 \cdot 8$.

ACID SUBSTANCES, BASIC SUBSTANCES, AND SALTS.

144. Some information concerning acids and salts has been already given in speaking of hydrochloric acid and the chlorides (**105–108**). This information may now be extended by making some experiments on the general properties of acid and basic substances, and of salts, and by learning methods for their quantitative measurement.

Certain changes in colour are produced with vegetable or other colouring matters and are made use of in these processes, and the coloured substances which undergo these changes are termed 'indicators.'

145. Indicators.—The use of two indicators, litmus and turmeric, may be described here. Certain substances have the power of altering the colour of these vegetable bodies. Litmus and turmeric are accordingly 'indicators' of the presence of these substances.

For instance, acid substances, including many free acids, turn blue litmus red, but do not alter the colour of turmeric. Alkaline substances, including many basic substances, turn red litmus blue and yellow turmeric brown.

By the careful addition of an acid to an alkali, a neutral substance can be produced, which causes neither litmus nor turmeric to change in colour.

Other colouring matters may be used as indicators instead of litmus and turmeric. Perhaps the most useful of these is methyl-orange; this substance is yellow in alkaline solution, but its colour changes to red when the solution is acidified.

The three varieties of substances, the soluble acids, bases, and salts, which give respectively acid, alkaline, and neutral reactions, may be taken as representatives of three classes of substances.

Expt. 178.—**Properties of Acids.**—Try the effect of the common dilute acids upon each of the following substances, and tabulate the results which are obtained.

(*a*) Blue litmus solution, or paper.
(*b*) Red litmus solution, or paper.
(*c*) Yellow turmeric paper.
(*d*) Methyl-orange solution.
(*e*) Sodium hydroxide solution coloured by litmus.
(*f*) Solid sodium carbonate.
(*g*) Metallic zinc.

Expt. 179.—**Properties of Bases.**—Try the effect of solution of sodium hydroxide and of solution of calcium hydroxide separately upon each of the following substances, and tabulate the results which are obtained.

(*a*) Red litmus solution, or paper.
(*b*) Blue litmus solution, or paper.
(*c*) Yellow turmeric paper.
(*d*) Methyl-orange solution acidified with one drop of acid.
(*e*) Dilute sulphuric acid coloured by litmus.

Expt. 180.—**Properties of Salts.**—Try the effect of adding solutions of sodium sulphate, of sodium carbonate, and of alum separately to each of the following substances, and note down the results obtained.

(*a*) Blue litmus solution, or paper.
(*b*) Red litmus solution, or paper.
(*c*) Yellow turmeric paper.
(*d*) Methyl-orange solution.
(*e*) Solid sodium carbonate.

It will be found that acids will change blue litmus to red, will alter the reaction of an alkali, will decompose carbonates with evolution of carbon dioxide, and will act upon zinc with evolution of hydrogen. Most of these properties are common to acids and distinguish this class of compounds from others.

It will be found that bases will show an alkaline reaction with test-papers, and will alter the reaction of acids, either neutralising or reversing the reaction according to the proportion of base which is added. It may be remarked that most bases are oxides or hydroxides. Many of them are insoluble in water, and accordingly cannot produce any reaction on test-paper. The reaction of a base is *never acid.*

On the other hand, neutral salts will cause no change of colour in the indicators, neither will they evolve carbon dioxide from carbonates. Many salts, however, possess an acid reaction, and the solution of some of them even causes carbon dioxide to be evolved from carbonates, and hydrogen to be evolved by zinc: alum is an example of such a salt. Other salts are alkaline in reaction and show the general character of bases: sodium carbonate is an example of a salt with alkaline reaction.

ACIDIMETRY AND ALKALIMETRY.

146. Estimation of Acids and Alkalis by Measurement of liquid Volume.—The volume of the solution of an acid which is required to neutralise a certain volume of the solution of an alkali may be ascertained by applying the information furnished in the preceding paragraph.

The usual method consists in measuring an exact volume of one liquid, say of the alkali, by means of a graduated cylinder (8) or pipette (9), and then gradually dropping the acid liquid into this from a burette (10) until the resultant liquid is neutral.

In order that the exact point of neutrality may be easily seen, a few drops of litmus solution are added to the alkaline solution, and the acid liquid is then dropped in until the blue colour of the liquid just changes to red. Methyl-orange solution may be used instead of litmus: the colour will then change from yellow to red just as neutralisation is reached.

The weights of acid and of alkali in solution, which are equivalent to each other, may be thus easily ascertained if the liquids are of known strength and their volumes are measured with care.

A few simple experiments will serve to illustrate this process.

EXPT. 181.—**Preparation of Standard Sulphuric acid Solution containing 20 grams of Acid per litre.**—Weigh 20 grams of pure concentrated sulphuric acid (H_2SO_4) in a weighed beaker. Pour this into a beaker containing about 500 c.c. of water, mix the liquids well, then rinse out the small beaker and add the washings to the liquid in the larger beaker. As soon as the liquid is cold transfer it to a stoppered litre cylinder, washing out the beaker and adding the washings to the liquid in the cylinder. Finally dilute the liquid to the litre-mark by the cautious addition of water, close the cylinder, and mix its contents thoroughly by shaking. The acid solution will now be ready for use.

Note that 20 grams of H_2SO_4 have been diluted to one litre or 1000 c.c., and that each c.c. of the liquid therefore contains $\frac{20}{1000} = 0{\cdot}02$ gram of H_2SO_4.

EXPT. 182.—**Preparation of Standard Potassium hydroxide Solution.**—Next weigh out quickly 20 grams of solid potassium hydroxide (KHO), avoiding contact of the caustic substance with the fingers or with the scale-pan. Dissolve this in a small quantity of water, and dilute the solution to a litre as in the preceding experiment. Each c.c. of this solution will contain 0·02 gram of KHO.

EXPT. 183.—**Determination of the Volume of the Sulphuric acid which is required to Neutralise various Volumes of the Potassium hydroxide Solution.**—Measure out 20 c.c. of the potassium hydroxide solution into an

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eight-ounce beaker by means of a pipette (9), and add to it a little water and a few drops of litmus solution.

Rinse out a burette (10) with a little of the acid solution, and then fill it to the zero mark with the same solution. Now add the acid solution gradually to the contents of the beaker, stirring continuously, until the blue colour of the litmus *just* changes to red. Read off the number of c.c. of acid which have been required for this purpose. A repetition of the process with 20 c.c. of fresh KHO solution should yield a result not differing by more than 0·2 c.c. from the first one.

Now measure out separately 40 c.c. and 60 c.c. of the alkaline solution into two beakers, and add the acid as before until each portion is exactly neutral. It will be found that these portions of the alkaline solution will require respectively double and treble the quantity of acid which was required by the 20 c.c. of the alkaline solution. Hence the proportion by volume of the acid solution to the alkaline solution is the same whatever quantity of the alkaline solution is neutralised.

Now calculate the weight of H_2SO_4 which would be required to neutralise one gram of KOH (147).

147. Calculation of the weight of Sulphuric acid which is required to neutralise a given weight of Potassium hydroxide.—Since weighed quantities of acid and alkali were used in preparing the acid and alkaline solutions, it is possible to calculate the *weight* of acid which has been required to neutralise a given weight of the alkali.

Example.—In a special determination 20 c.c. of the potassium hydroxide solution required for neutralisation 17·8 c.c. of the standard sulphuric acid.

Now each c.c. of these solutions contains 0·02 gram of acid or of alkali,

Therefore $20 \times 0{\cdot}02 = 0{\cdot}4$ gram of KHO requires $17{\cdot}8 \times 0{\cdot}02 = 0{\cdot}356$ gram of H_2SO_4 for its neutralisation.

Therefore one gram of KHO would require $\frac{0{\cdot}356 \times 1}{0{\cdot}4} = 0{\cdot}89$ gram of H_2SO_4 for its neutralisation.

Expt. 184.—**Determination of the Weight of Sulphuric acid required to Neutralise a given Weight of Sodium hydroxide Solution.**—Weigh out 20 grams of sodium hydroxide (NaHO); dissolve this in water, and make the solution up to a litre, as was done in the case of potassium hydroxide (Expt. 182). Measure out 20 c.c. of this solution, and exactly neutralise it with the standard sulphuric acid, using litmus as an indicator. Calculate as before (147) the weight of the acid which would be required to neutralise one gram of NaHO.

Example.—In an actual experiment it was found that 20 c.c. of sodium hydroxide solution were neutralised by 23·6 c.c. of the sulphuric acid solution. Therefore, $20 \times 0·02 = 0·4$ grams of NaHO required for neutralisation $23·6 \times 0·02 = 0·472$ grams of H_2SO_4, and one gram of NaHO would require 1·18 gram of H_2SO_4.

Expt. 185.—**Sulphuric acid and Sodium Hydrogen carbonate Solution.**—Weigh out 20 grams of sodium bicarbonate ($NaHCO_3$), dissolve this in cold water, and dilute the solution to a litre with water. Neutralise 20 c.c. of this solution with the sulphuric acid solution, using methyl-orange as an indicator.

Example.—In an actual experiment, 20 c.c. of the bicarbonate solution required 12·0 c.c. of the sulphuric acid for neutralisation. Accordingly, 0·4 gram of $NaHCO_3$ neutralises 0·24 gram of H_2SO_4, and one gram of $NaHCO_3$ would require 0·6 gram of H_2SO_4 for neutralisation.

Expt. 186.—**Sulphuric acid and Sodium Diborate (Borax) Solution.**—Dissolve 20 grams of crystallised borax ($Na_2B_4O_7 . 10H_2O$) in water and make the solution up to a litre. Titrate 20 c.c. of this solution with the sulphuric acid solution, using methyl-orange as an indicator.

Example.—In an actual experiment, 20 c.c. of the borax solution required 5·1 c.c. of the sulphuric acid solution for neutralisation. Therefore, $20 \times 0·02 = 0·4$ gram of crystallised borax requires for neutralisation $5·1 \times 0·02 = 0·102$ gram of H_2SO_4, and one gram of borax would require 0·26 gram of H_2SO_4.

148. **Equivalence of the Alkaline Solutions.**—According to the above experiments the following quantities of the

alkaline solutions are equal to each other in their power of neutralising a given volume of the acid.

20 c.c. of the KHO solution neutralise 17·8 c.c. of the H_2SO_4 solution, and 23·6 c.c. of the H_2SO_4 solution are neutralised by 20 c.c. of the NaHO solution. Therefore 17·8 c.c. of the H_2SO_4 solution would be neutralised by $\frac{20 \times 17{\cdot}8}{23{\cdot}6} = 15{\cdot}1$ c.c. of the NaHO solution.

In the same way it can be calculated that 17·8 c.c. of the H_2SO_4 solution would be neutralised by $\frac{20 \times 17{\cdot}8}{12{\cdot}0} = 29{\cdot}7$ c.c. of $NaHCO_3$ solution, and by $\frac{20 \times 17{\cdot}8}{5{\cdot}1} = 69{\cdot}8$ c.c. of the borax solution.

Accordingly, if the amount of acid neutralised by 20 c.c. of the KHO solution is taken as the standard, the equivalence, as regards neutralising power, of the following volumes of the alkaline solutions has been established:—20·0 c.c. of the KHO solution: 15·1 c.c. of the NaHO solution: 29·7 c.c. of the $NaHCO_3$ solution: and 69·8 c.c. of the borax solution. Or 0·4 gram of KHO, 0·302 gram of NaHO, 0·594 gram of $NaHCO_3$, and 1·396 gram of borax have the same neutralising power, since each of these quantities neutralises 0·356 gram of H_2SO_4.

149. Calculation of the Weights of Sulphuric acid which are equivalent to equal Weights of different Alkaline Substances.—If 20 c.c. of the solution of potassium hydroxide, of sodium hydroxide, and of sodium bicarbonate are used, the weights of the solids dissolved in each case will be the same, namely, 0·4 gram. It will be found that different volumes, and therefore different weights, of sulphuric acid are necessary for neutralising these equal weights. The ratios of these equal weights to the weights of acid will be found to be the same as the ratios of the weights shown in following equations.

	Ratios.
$H_2SO_4 + 2KHO = K_2SO_4 + 2H_2O$	H_2SO_4 KHO
98 112·3	0·873 : 1
$H_2SO_4 + 2NaHO = Na_2SO_4 + 2H_2O$	H_2SO_4 NaHO
98 80	1·23 : 1
$H_2SO_4 + 2NaHCO_3 = Na_2SO_4 + 2H_2O$	H_2SO_4 $NaHCO_3$
98 168 $+ 2CO_2$	0·58 : 1
$H_2SO_4 + Na_2B_4O_7.10H_2O = Na_2SO_4 + 4H_3BO_3$	H_2SO_4 Borax
98 382 $+ 5H_2O$	0·25 : 1

Note.—It will be noticed, however, that the experimental results given above do not quite agree with the theoretical values indicated by the equations. The difference is due (1) to the fact that some of the substances which are used in these experiments are not quite pure, and (2) to the slight errors which are necessarily involved in carrying out the experiments.

150. Use of Standard Solutions in determining Acids and Alkalis of unknown Strength.—It is evident that the weight of acid which is capable of neutralising a known weight of a particular alkali can be found both by experiment and by calculation. Hence if a solution of alkali of unknown strength is supplied the weight of alkali in solution can be experimentally determined by means of an acid solution of known strength, provided that the reaction between the acid and the alkali is known.

This is also the case when an acid of unknown strength is neutralised by an alkali of known strength.

Expt. 187.—**Determine the Strength of the dilute Acid on the Laboratory Bench**, taking 5 c.c. of the acid and using standard potassium hydroxide solution (40 grams per litre) in the burette.

Example.—5 c.c. of dilute HCl were measured out into a beaker, and were mixed with a few drops of methyl-orange; standard NaHO solution (1 c.c. = 0·04 gram) was then added until the acid was neutralised. It was found that 5 c.c. of the acid required 41 c.c. of the alkaline solution for its neutralisation.

The equation $NaHO + HCl = NaCl + H_2O$ shows that 40 grams of NaHO neutralise 36·45 grams of HCl.

Therefore one gram of NaHO will neutralise $\frac{36 \cdot 45}{40} = 0 \cdot 9112$ gram of HCl.

And 1 c.c. (=0·04 gram) neutralises $0 \cdot 9112 \times 0 \cdot 04 = 0 \cdot 0364$ gram of HCl.

Therefore 5 c.c. of the dilute HCl contain $41 \times 0 \cdot 0364 = 1 \cdot 4924$ gram of pure HCl, and one litre contains $1 \cdot 4924 \times 200 = 298 \cdot 5$ grams of HCl.

Expt. 188.—Determine the strength of the laboratory solutions of sodium carbonate and of potassium hydroxide by means of standard sulphuric acid (40 grams H_2SO_4 per litre).

MISCELLANEOUS EXAMPLES.

151. A number of examples are given below, which may be worked out with little or no assistance at this stage, from the experience which has been already obtained.

The outline of the procedure to be adopted in each case is given.

Preparation of Crystalline Salts.

152. The general method of procedure is as follows:—Dissolve as much as possible of the metal or other substance in a suitable quantity of the requisite acid, using heat if necessary. If the solution is not clear, either decant or filter it from the solid matter. Then concentrate the liquid by evaporation until the salt crystallises out as the liquid cools.

Describe the crystalline shape, the appearance, and other properties of the product obtained.

The following will be suitable examples:—

(1) **Zinc sulphate, $ZnSO_4.7H_2O$,** from dilute H_2SO_4 and metallic Zn.

(2) **Iron sulphate, $FeSO_4.7H_2O$,** from dilute H_2SO_4 and clean iron turnings.

(3) **Magnesium sulphate, $MgSO_4.7H_2O$,** from dilute H_2SO_4 and MgO.

(4) **Copper sulphate, $CuSO_4.5H_2O$,** from dilute H_2SO_4 and $CuCO_3$.

(5) **Copper nitrate, $Cu(NO_3)_2.6H_2O$,** from strong nitric acid diluted with an equal volume of water and copper turnings.

(6) **Lead nitrate, $Pb(NO_3)_2$,** from nitric acid of the above strength and metallic lead.

(7) **Potassium nitrate, KNO_3,** from dilute HNO_3 and KHO solution.

(8) **Tin Chloride, $SnCl_2.2H_2O$,** from strong hydrochloric acid, diluted with its own volume of water, and metallic tin.

(9) **Barium Chloride, $BaCl_2.2H_2O$,** from dilute HCl and $BaCO_3$.

PREPARATION OF SOLUBLE AND INSOLUBLE SALTS BY DOUBLE DECOMPOSITION.

153. A very useful exercise in precipitation and crystallisation consists in forming an insoluble salt and a crystallised soluble salt by double decomposition, from weighed quantities of two original salts. An example will make this clear.

EXPT. 189.—**To form $BaSO_4$ and $CuCl_2$ by double decomposition.**—Weigh out quantities of crystallised copper sulphate ($CuSO_4.5H_2O$) and barium chloride ($BaCl_2.2H_2O$), such as are required by the following equation.

$$\underset{244\cdot3}{BaCl_2.\,2H_2O} + \underset{249\cdot6}{CuSO_4.5H_2O} = \underset{233\cdot4}{BaSO_4} + \underset{170\cdot5}{CuCl_2.2H_2O} + 5H_2O$$

As the above weights in grams derived from the equation will be too large, the numbers may be divided by 40. The weights to be taken will therefore be

6·1 grams of $BaCl_2.2H_2O$ and 6·2 grams of $CuSO_4.5H_2O$.

Dissolve each of the weighed salts separately in about 50 c.c. of distilled water, and mix the two solutions together.

Separate the precipitate from the liquid by filtration, and wash (**188**), dry (**189**), and weigh the $BaSO_4$. It should amount to $233 \div 40 = 5{\cdot}8$ grams.

Collect the filtrate and the first washings and evaporate the liquid until it deposits crystals of $CuCl_2.2H_2O$ on cooling. Note the appearance of the crystals. Then dry them by pressing them gently between filter-paper, and weigh them. Compare the weight found with the weight which should be found as calculated from the equation. Since some $CuCl_2$ will always remain in solution, the weight of the crystals will probably be only about two-thirds of the amount calculated from the equation.

Similar preparations may be made from the following substances :—

(1) **Lead nitrate and Potassium chromate** :—

$$Pb(NO_3)_2 + K_2CrO_4 = PbCrO_4 + 2KNO_3.$$

(2) **Lead nitrate and Copper sulphate** :—

$$Pb(NO_3)_2 + CuSO_4.5H_2O + H_2O = PbSO_4 + Cu(NO_3)_2.6H_2O.$$

(3) **Copper sulphate and Potassium ferrocyanide** :—

$$2CuSO_4.5H_2O + K_4FeCy_6.3H_2O = Cu_2FeCy_6 + 2K_2SO_4 + 13H_2O.$$

ESTIMATION OF THE SOLUBILITY OF SUBSTANCES IN WATER.

154. The determination of the solubility of a substance in water at different temperatures, forms a useful exercise. The results should be plotted out as a curve on squared paper, the temperatures forming the abscissæ, and the percentage weight dissolved forming the ordinates, Expt. 190.—Determine the solubility of potassium chlorate and of potassium dichromate at the temperatures of 15° C., 20° C., 25° C., 30° C., and 40° C. by the method described in paragraph 66. Plot out a curve on one tenth-inch squared paper, the temperatures being placed horizontally and the percentages dissolved vertically.

Conversion of Nitrates and Carbonates into Oxides.

155. Many nitrates and carbonates of metals are converted into oxides when they are strongly heated. By weighing out accurately between one and two grams of the salt, and heating it in a crucible until the weight remains constant, the relation by weight of the salt to the oxide can be determined.

Expt. 191.—Ascertain the weight of oxide which remains when 1·5 grams of each of the following salts are heated :—Lead nitrate, barium nitrate, copper carbonate, and zinc carbonate.

Effect of Heating a Substance alone, in Air, and in Hydrogen.

156. The study of the action of heat upon a substance out of contact with air, in a current of air, and in a current of dry hydrogen, will furnish a useful series of exercises, particularly if the reaction is studied quantitatively as well as qualitatively.

Expt. 192.—Try the effect of heating about two grams of the following substances :—Red lead, copper oxide, coke, ferric oxide, iron pyrites, and manganese dioxide.

(1) In a narrow combustion-tube closed at one end.
(2) In a tube open at both ends, through which a current of air is passing.
(3) In a current of hydrogen which has been previously dried.

DETERMINATION OF THE WEIGHT OF A LITRE OF CARBON DIOXIDE GAS.

157. If a carbonate is heated under suitable conditions with an excess of acid, the loss of weight is equal to the weight of carbon dioxide which was in the carbonate (87). The volume of the gas which is evolved during this change may also be ascertained (86). From the two results thus obtained the weight of one litre of carbon dioxide gas may be calculated.

EXPT. 193.—Obtain the weight of a litre of carbon dioxide gas by two experiments, using for this purpose first calcium carbonate ($CaCO_3$), and then sodium carbonate crystals ($Na_2CO_3.10H_2O$).

DETERMINATION OF THE COMPOSITION OF WATER BY WEIGHT.

158. The composition of water has been studied in paragraphs 52, 53, and it was shown that water is a compound of oxygen and hydrogen. The most convenient method of ascertaining the proportion by weight of these two elements in water depends upon the action of pure dry hydrogen upon copper oxide at a red heat. The hydrogen combines with the oxygen to form water and the copper oxide is reduced to metallic copper :—$H_2 + CuO = H_2O + Cu$.

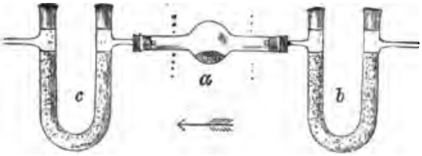
The water is collected and weighed, and the decrease in weight of the copper oxide is ascertained. The weight of the oxygen which has combined with the hydrogen to form the water is thus obtained. From these data the relation by weight of the oxygen to the hydrogen in water can be calculated.

EXPT. 194.—Arrange the apparatus for the preparation of pure hydrogen by the action of *pure* hydrochloric acid upon zinc free from As and Sb (fig. 34, p. 56), allowing the gas to pass through a bottle containing strong H_2SO_4 (fig. 54, p. 97).

Prepare a U-tube, (*b*) fig. 60, or (*b*) fig. 36, p. 58, as follows. Break up some pumice-stone into pieces about the size of hemp-seeds, and nearly fill the U-tube with these fragments. Glass beads will do equally well. Pour sufficient strong sulphuric acid into the tube to cover the contents, and then incline the tube so as to drain away most of the acid. Remove the acid completely from the necks of the tube by means of a wet cloth or filter-paper, and insert two well-fitting perforated corks. The U-tube may be filled with granulated calcium chloride, instead of with fragments wetted with H_2SO_4. This U-tube is used for the purpose of drying the hydrogen gas.

In fig. 60 a bulb tube of hard glass (*a*) is shown which contains five grams of pure copper oxide. The oxide must have been recently heated to redness for some time in a stream of air and then allowed to cool. The tube is supported on the ring of a retort-stand. The apparatus for the absorption of the water is another U-tube (*c*), prepared as above and provided with a bulb near (*a*), which is not shown in the figure.

FIG. 60.



FORMATION OF WATER FROM HYDROGEN AND COPPER OXIDE.

This bulb serves to catch the greater part of the water which is formed during the experiment. The remainder of the water vapour is absorbed by the sulphuric acid, or $CaCl_2$, in the other portions of the tube (*c*).

The dotted lines show two perforated discs of asbestos-board, which prevent the corks from being charred, and enable the heating to be controlled.

Now pass a slow current of hydrogen through the whole apparatus, until the air has been entirely displaced, and heat the copper oxide to dull redness in the stream of hydrogen until the black colour of the oxide has disappeared.

First prove the apparatus to be air-tight. Then weigh the tube containing the copper oxide: weigh also the bulbed absorption U-tube (*c*): and connect the parts of the apparatus together as is shown in the figure.

Drive over any water which has condensed in the combustion-tube (*a*), into the absorption-tube (*c*) by heating it with a flame. Then allow the apparatus to cool, and again weigh the copper oxide tube and the absorption-tube (*c*).

The gain in weight of the absorption-tube (*c*), which represents the amount of water formed, will bear to the loss of weight of the copper oxide tube, which represents the weight of oxygen in the water, the proportion of 9 to 8, if the experiment has been carefully performed.

This result proves that the water contains eight-ninths its weight of oxygen and one-ninth its weight of hydrogen.

PART II.

QUALITATIVE CHEMICAL ANALYSIS.

PART II.

SECTION XI.

ANALYTICAL OPERATIONS.

Each student should work independently through the Sections on Chemical Analysis.

In all analytical work the *water* used must be *distilled water*, which may be conveniently kept in the wash-bottle.

Most of the operations referred to in this Section are used solely in analytical work, but many ordinary analytical operations have been already fully described and illustrated in Section V. These include **Distillation, Solution, Evaporation, Crystallisation, Precipitation, Filtration, and Decantation.** They will not be further described.

180. Ignition.—Solid substances are frequently heated strongly, or *ignited*, in order to study the effect of heat upon them.

The following are the most important changes which may take place when a substance is ignited:—

a. The substance changes colour.

b. Water is given off, which may be acid or alkaline in its reaction to test-paper (191).

c. The substance melts or fuses.

d. A sublimate is formed, which may be coloured or colourless.

e. A gas is given off, probably one of those which have been already studied in Sections IV. to IX.

Expt. 210.—Heat a little white zinc oxide in a small ignition-tube. The colour will be *lemon yellow* while the oxide is hot, but will change to *white* again when it has cooled. These changes of colour are characteristic of zinc oxide.

Expt. 211.—Heat a small piece of ammonium chloride in a small test-tube. The ammonium chloride will pass off as vapour, or *volatilise*, and the vapour will cool in the upper part of the tube and form a coating or *sublimate*. The substance is accordingly said to be *volatile*.

Expt. 212.—Heat a little powdered lead nitrate in an ignition-tube.

The substance will crackle or *decrepitate*, and reddish-coloured gas will be given off, which causes a glowing match to burst into flame. The residual substance will change colour to yellow, and then to red, and will finally fuse. It will become yellow again as it cools.

Expt. 213.—Heat each of the following substances in an ignition-tube, and carefully note down every change which occurs :—Red lead, copper sulphate, arsenic trioxide, copper carbonate, and manganese oxalate.

181. Heating with Fused Reagents.—A substance is often heated with solid reagents which have been *fused*, in order to bring about chemical changes which yield a characteristic result.

Expt. 214.—Fuse a little powdered sodium carbonate and potassium nitrate upon a piece of platinum-foil, by holding the foil with crucible-tongs, and heating its under surface by the tip of the Bunsen-flame or of the blowpipe-flame. Now add a minute quantity of manganese dioxide, and maintain the mass in a fused condition for a short time; then allow it to cool. It will be *bluish-green* when it has cooled. This colour is an indication of the presence of manganese.

EXPT. 215.—Treat *a little* chromium sesquioxide in a similar manner, and note down the results which are obtained.

182. Flame Reactions.—The flame reactions described in paragraphs 182 to 186, differ from those which have been already described (180, 181), since the gases of the flame are allowed to take part in the change.

In a well-formed blowpipe-flame (23) two conical portions are readily seen: the *inner flame*, which is blue and pointed; and around and beyond this an almost colourless flame, known as the *outer flame*. A similar distinction can be observed in the Bunsen-flame.

The inner flame is often called the *reducing* or *deoxidising flame*, because it separates or reduces metals from many of their compounds, and in general deoxidises substances. This is due to the action of the hot but unburnt hydrogen, carbon monoxide, and hydrocarbons which are present in this part of the flame.

The outer flame is often called the *oxidising flame*, because it changes metals into their oxides, and oxidises many other substances by exposing them to oxygen at a high temperature.

The results obtained in the following processes will usually vary according to the part of the flame in which the substance is heated.

183. Fusion with Borax: the Borax-bead.—When certain metals or their compounds are heated with fused borax they are converted into coloured borates. The colour of each of these borates is characteristic, either in itself or in the change which it undergoes in the outer or inner blowpipe-flame.

EXPT. 216.—Moisten the loop of a piece of mounted platinum-wire (25), and dip it into a little powdered borax. Then hold the loop with the adhering powder in the outer blowpipe-flame until the borax is melted. If the bead thus formed does not fill the loop, dip the melted bead again into the borax, and melt the borax once more. The bead must be perfectly colourless and transparent when it is cold.

Moisten the clear cold borax-bead, and take up on its surface a *few particles only* of finely powdered manganese dioxide. Fuse these particles into the bead, by heating it for a short time in the tip of the *outer* blowpipe-flame. The bead will appear *purple or amethyst red* while it is hot, and this colour will not alter as the bead cools.

Now heat the bead again for some time in the tip of the *inner* blowpipe-flame. It will become colourless, and will remain so when it is cold. This change of colour in the borax-bead characterises manganese compounds.

In a first attempt this bead is often opaque, since too much manganese oxide has been used. If this should happen, a portion of the fused bead should be shaken off and replaced by fresh borax, and the process should be repeated until the coloured bead is transparent.

As soon as the experiment is finished, the fused bead may be removed by giving the wire a sudden jerk: or the cold bead may be crushed on a hard flat surface by a sharp blow with the pestle.

Two mounted platinum-wires should be kept with their loops immersed in dilute hydrochloric acid; they may then be rapidly cleansed by washing them with water before they are used.

Expt. 217.—Heat a small quantity of each of the following substances in a freshly made borax-bead, and note the appearance of the bead after it has been heated in the outer and in the inner flame:—

Chromium oxide: ferric oxide: copper sulphate.

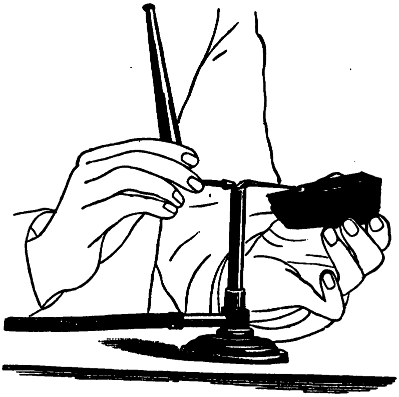
184. Ignition on Charcoal in the Blowpipe-flame.—A substance is usually ignited on charcoal in order to detect the presence of the heavier metals. These are separated from the substance by the action of the inner blowpipe-flame and of the red-hot charcoal. Fused reagents are often employed to assist the separation.

Some metals are separated in the vaporous condition, and the vapour burns as it leaves the charcoal; the metallic oxides formed by the combustion are deposited as *incrustations* on the charcoal. The white ash which is left by the combustion of the charcoal must not be mistaken for such an incrustation.

The compounds of many metals do not yield their metals under the above conditions; but occasionally a smell, or a change of colour or condition, occurs which is characteristic. Thus the salts of sodium and potassium fuse and sink into the charcoal, colouring the flame with a characteristic tint.

Expt. 218.—Select a sound piece of wood-charcoal, which does not crackle when it is heated in the blowpipe-flame. Scoop out a small hollow near one end with a knife or with a charcoal-borer. Place in this cavity a small quantity of powdered lead acetate and of sodium carbonate, and mix the powders together in the cavity with the point of a penknife. Heat this mixture in the inner blowpipe-flame, allowing the flame to play over the surface of the charcoal (fig. 61).

Fig. 61.



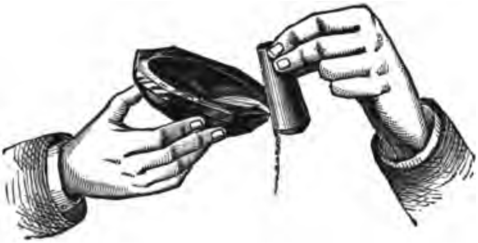
Heating upon Charcoal.

Bright globules of lead will soon be visible in the cavity; and when the charcoal is removed from the flame, the surface surrounding the cavity will be found to be coloured with a yellow *incrustation.*

One of the globules of metal should be taken off the charcoal with the point of a penknife, and struck smartly with the pestle on the bottom of the inverted mortar. It will flatten out into a cake without being crushed into powder, showing that the metal is *malleable* and not *brittle.*

Another globule should be cleansed by rolling it on moistened filter-paper under the tip of the finger; if it is then transfixed on the point of a penknife-blade, it will be found to mark paper as a black-lead pencil does.

FIG. 62.



WASHING BY LEVIGATION.

No other metallic compounds give a yellow incrustation and malleable globules which are soft enough to mark paper; this test therefore serves to detect the presence of lead.

EXPT. 219.—Heat each of the following substances with a mixture of sodium carbonate and potassium cyanide on charcoal, and note down the results which are obtained:—

Tartar emetic: tin dioxide: and bismuth oxi-nitrate.

185. Washing and Levigation of the Residue which is left on the Charcoal.—The metallic globules which are produced in the preceding experiments are usually easily visible either with or without the aid of a lens. They are also easily detached from the charcoal.

But when the metal separates in the form of fine metallic particles or scales it can often only be separated and detected after the cool saline residue has been detached from the charcoal and crushed in a mortar or in a porcelain-dish with a little water, so as to dissolve the alkaline salts. The particles are usually more readily seen, if the water is stirred up and is at once quickly decanted down the wet pestle, held against the edge of the vessel (fig. 62). The light particles of charcoal are thus poured off, suspended in the saline solution, while the heavier and insoluble metallic powders are left in the vessel. If this process of *levigation* is repeated several times, the metal may be entirely freed from soluble substances and from charcoal, and may then be further examined by a lens or magnet or by other tests.

Expt. 220.—Fuse a little of each of the following substances with a mixture of sodium carbonate and potassium cyanide on charcoal in the inner blowpipe-flame, and wash the residue as is directed above, carefully recording the results:—

Copper sulphate: ferrous sulphate: nickel sulphate.

186. Flame Colorations.—When a sufficiently volatile substance is heated in a non-luminous flame it emits vapour which often imparts a characteristic colour to the flame. The blowpipe-flame may be employed to obtain this coloration, but a Bunsen-flame usually answers the purpose well.

Expt. 221.—Hold a platinum-wire loop (**25**) in the inner blowpipe-flame; if it is clean, it will become red-hot without colouring the flame. If any colour is produced, the wire must be wetted with strong hydrochloric acid, and then heated in the blowpipe-flame until it no longer colours the flame. If the loop cannot be cleansed in this way, the end of the wire must be cut off and a fresh loop must be formed.

Dip the clean wire loop into a little sodium chloride solution, and hold it in the blowpipe-flame and in the Bunsen-flame. Each of these flames will be coloured *bright yellow*.

This yellow coloration is given to the flame only by sodium and by its volatile compounds. If the yellow flame is examined through a long glass prism filled with indigo-solution no yellow coloration will be seen.

Remove all the sodium chloride from the platinum-wire loop, by heating it strongly in the tip of the blowpipe-flame until it ceases to impart any colour to the flame. Then dip the wire loop into some solution of *pure* potassium nitrate. This will impart to the flame a *pale lilac* colour, which is characteristic of potassium and its compounds. If this coloration is examined through the indigo-prism, it will appear *crimson*.

Expt. 222.—Dip the wire loop into a mixture of a few drops of the sodium chloride and of the potassium nitrate solutions. When the loop is held in the flame the sodium coloration only will be seen. If this coloration is then viewed through the indigo-prism, the crimson potassium coloration will be seen distinctly. By means of the indigo-prism the potassium coloration is therefore discoverable, even when it is concealed by that of sodium.

Expt. 223.—Heat a little of each of the following substances on platinum-wire in the Bunsen-flame and note down the results which are obtained :—

Barium chloride : calcium chloride : copper sulphate : and strontium nitrate.

187. Analytical Operations with Precipitates.—The operations of precipitation, filtration, and decantation have been described in paragraphs 69–71.

The further treatment which a precipitate may require during analysis is described below (**188–190**).

188. Washing Precipitates.—A precipitate, which has been separated from the liquid in which it was suspended, must usually be *washed* until it is perfectly free from the adhering saline solution.

The precipitate may be washed upon the filter, if it has been separated from the liquid by filtration. If it has been separated by decantation, it is usually washed by decantation.

Expt. 224.—**Washing by Filtration.**—Mix a little ammonium carbonate solution with some barium chloride solution, and filter off the resulting precipitate. Blow into the funnel containing the filter and the barium carbonate precipitate a fine stream of hot distilled water from the wash-bottle, directing the jet so as to stir up the precipitate. Fill the paper with water to within a short distance from its edge, and as soon as this water has completely run through, fill the filter again in the same way. Repeat the process three or four times.

The precipitate and filter will then usually be free from everything which is soluble in water.

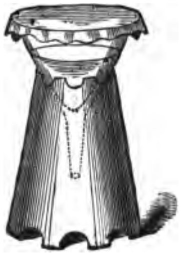
Ascertain whether the washing is complete, by collecting the *last few drops* of the washing-water in a clean test-tube, and adding to it a drop of silver nitrate solution. If no turbidity is produced, the absence of dissolved chloride is proved. If turbidity appears, the washing must be continued and the washing-water must be again tested. These processes are repeated until no turbidity is produced.

EXPT. 225.—**Washing by Decantation.**—The precipitate of lead carbonate obtained by adding ammonium carbonate solution to lead acetate solution may be washed by decantation. For this purpose the precipitate is shaken, stirred, or boiled with hot water, and is then allowed to settle. As soon as the water has become clear, it is poured off in the manner shown in fig. 45 (p. 74). If this washing process is repeated several times with fresh portions of boiling distilled water, the precipitate may be entirely freed from the adhering saline solution. As the washing-water is poured off, it is tested occasionally with turmeric paper, to ascertain when it ceases to turn the paper brown, and is therefore free from sodium carbonate. As soon as this is the case the precipitate has been thoroughly washed.

It is usual to ascertain when the washing of a precipitate is complete by testing the washing-water for that soluble substance which may be most readily detected, as is seen in the above examples.

189. Drying Precipitates.—A precipitate is dried by placing the funnel in a hollow tin cone or cylinder, called a *filter-dryer* (fig. 63). This is supported either on a piece of iron wire-gauze over a small flame, or upon a heated iron plate or sand-bath. The funnel is thus exposed to a current of hot air, which rapidly dries the filter and precipitate. Great care must be taken not to char the filter.

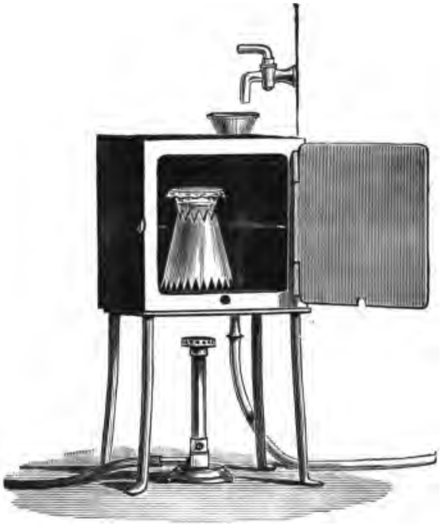
FIG. 63.



THE FILTER-DRYER.

A precipitate may be more rapidly dried by draining it in the funnel, and then removing the filter and spreading it upon a piece of wire-gauze. The gauze is then supported above a small flame from a rose-burner, at such a distance as to prevent any risk of charring the paper.

FIG. 64.



THE STEAM-OVEN.

The drying of a moist precipitate may be effected slowly and safely by placing it inside a steam-oven (fig. 64). All risk of charring the paper is thus avoided.

A precipitate may be partially dried by opening out the filter which contains it upon several dry filter-papers, and allowing them to absorb the water. This process may precede those already mentioned.

190. Removal of Precipitates from the Filter-paper.

1. **Partial Removal.**—A small quantity of a moist precipitate may be most readily taken from a filter by dipping the end of a glass rod into it.

A sufficient quantity of a bulky precipitate may often be taken out of the filter by means of a glass or porcelain spatula.

2. **Complete Removal.**—If the precipitate is to be removed from the filter as completely as possible, one of the following methods may be employed.

a. A hole may be made in the bottom of the filter by means of a slender glass rod, which is pushed down through the neck of the funnel. The precipitate is then easily washed down into a vessel beneath by a fine stream of water, or other suitable liquid, from the wash-bottle.

b. If it is undesirable to add a liquid to the precipitate upon the filter, the filter and precipitate are allowed to drain for a short time, and are then removed together from the funnel. The precipitate may be further drained, if necessary, by laying the filter on several dry filter-papers. The filter is then spread out upon a flat piece of glass, and the precipitate is carefully scraped off with a glass rod pressed flat upon the paper, or with a small spatula. This method is usually the most imperfect, but is frequently the best for other reasons.

c. A precipitate may also be removed from the filter, by means of the liquid with which it is to be treated or dissolved, in the following manner. The funnel is supported over a suitable vessel. The liquid is poured into the filter and is quickly stirred up with the precipitate by means of a thin glass rod. The liquid is poured again into the filter if any precipitate remains, and this process is repeated until all the precipitate is removed. Or the bottom of the filter may be pushed out through the neck of the funnel by the glass rod, and the liquid carrying the precipitate may be allowed to run through.

191. **Use of Test-papers.**—The two vegetable colouring substances, *litmus* and *turmeric*, undergo marked changes of colour when they are acted upon by certain substances. Slips of paper which have been stained with a solution of

one of these colouring substances are known as *test-papers*, and are easily distinguished by their colour.

Expt. 226.—Try the effect of the following substances upon blue and red litmus papers, and upon yellow turmeric paper, thus proving the truth of the following statements.

Hydrochloric acid reddens blue litmus, but does not alter the colour of red litmus or of turmeric: hence it is said to possess an *acid reaction.*

Ammonium hydrate solution does not affect the colour of blue litmus, but turns red litmus blue and turmeric brown: it is accordingly stated to show an *alkaline reaction.*

Barium chloride solution does not affect the colour of any one of the test-papers: its reaction is therefore termed *neutral.*

Each of these liquids is typical of a large class of substances in so far as its behaviour with these test-papers is concerned.

Expt. 227.—Add solution of ammonium hydrate, drop by drop and with constant stirring, to a little dilute hydrochloric acid, touching the wet end of the stirring-rod occasionally against pieces of blue and red litmus paper. By proceeding cautiously, a point may be reached at which the liquid does not affect the colour of either the blue or the red paper: the acid is then said to have been *neutralised.*

When another drop of ammonium hydrate is added, the liquid will acquire an alkaline reaction: the alkaline solution is then said to have been added *in excess.*

Conversely, an alkaline solution may be neutralised with an acid, and may be *acidified* or mixed with *acid in excess.*

The process of neutralising a liquid is most readily completed by the addition of very dilute acid or alkaline solution towards the end of the process. The addition of acid or of alkali in excess is thus more easily avoided.

SECTION XII.

ANALYTICAL REACTIONS.

INTRODUCTORY REMARKS ON ANALYTICAL CLASSIFICATION.

The Analytical Groups of the metals are tabulated in the next paragraph.

The acid-radicles are grouped in a similar way as far as possible.

200. Analytical Classification.—The systematic name of an inorganic chemical compound usually consists of the name of the metal or hydrogen which it contains, associated with the name of the element or radicle with which the metal or hydrogen is combined. Thus in the case of the salt sodium sulphate, the name of the metal *sodium* is associated with that of the *sulphate* acid-radicle. The latter portion of the name, in fact, usually denotes an acid-radicle, but occasionally hydroxyl or oxygen or some other element.

The hydrogen salts of acid-radicles are called *acids*. Thus the acid-radicles Cl, SO_4, PO_4, form the hydrogen salts or acids, HCl, H_2SO_4, H_3PO_4, and the metallic salts KCl, $MgSO_4$, Na_3PO_4. Some of these acid-radicles are elements, but most of them are *compound radicles*.

The above division of the name of a chemical compound generally corresponds to the portions of the compound substance which are separately detected during its analysis. For analytical purposes substances are accordingly classed as *Metals* and *Acid-radicles*.

201. Analytical Reactions and Reagents.—The usual object of qualitative analysis is to discover what element, elements, or sometimes what compound radicles a substance of unknown composition contains. Occasionally, however, the object is limited to ascertaining whether any particular element or radicle is present in a given substance.

For ordinary analytical purposes certain marked peculiarities of behaviour of each element or radicle under special conditions are selected, and by these *tests* or *reactions* its presence is detected.

The tests or reactions usually depend upon the changes which a substance undergoes when it is heated under various conditions, or when it is mixed with certain liquid or solid substances.

Such substances, added to produce any analytical test or reaction, are called *reagents.* Some reagents serve for detecting one element or compound only, and are therefore called *special reagents*; others separate or detect a group of elements or compounds, and are called *group reagents* or *general reagents.*

202. Analytical Groups of Metals.—The metals are separated into five principal Groups by their different behaviour with certain general reagents. Two of these Groups are further subdivided into two Subgroups. Each Group receives a distinctive name either from its place in the system, or from some conspicuous or important member of the Group, or from the general reagent which is used to precipitate the Group. Thus we speak indifferently of the Fourth Group, the Barium Group, or the Ammonium Carbonate Group.

203. ANALYTICAL CLASSIFICATION OF THE METALS.

GROUP I.		GROUP II.			
Metals whose chlorides are insoluble in water and in dilute acid, and which are therefore precipitated from their solutions by hydrochloric acid.		Metals whose sulphides are insoluble in dilute acid, and which are therefore precipitated from *acid* solutions by hydrogen sulphide.			
Hydrochloric acid, or Silver Group.		**Hydrogen sulphide Group.**			
		GROUP II.A. **Copper Group.** Sulphides insoluble in NaHO and in Am_2S.		GROUP II.B. **Arsenic Group.** Sulphides soluble in NaHO and in Am_2S.	
[Lead	Pb]	Mercuricum	Hg''	Tin	Sn
Silver	Ag	[Lead	Pb]	Arsenic	As
Mercurosum	Hg'	Bismuth	Bi	Antimony	Sb
		Copper	Cu		
		Cadmium	Cd		

GROUP III.				GROUP IV.		GROUP V.	
Metals which are precipitated by ammonium hydrate as hydrates from their solutions containing ammonium chloride, or as sulphides or hydrates when ammonium sulphides or hydrogen sulphide is subsequently added.				Metals whose carbonates are precipitated by addition of ammonium carbonate in the presence of ammonium chloride.		Metals whose solutions are not precipitated by any of the foregoing group-reagents in the presence of ammonium chloride.	
Ammonium hydrate and Ammonium sulphide Group.				**Ammonium carbonate, or Barium Group.**		**Potassium Group.**	
GROUP III.A. **Ammonium hydrate, or Iron Group.**		GROUP III.B. **Ammonium sulphide, or Zinc Group.**					
Aluminium	Al	Zinc	Zn	Barium	Ba	Ammonium	NH_4
Iron	Fe	Manganese	Mn	Strontium	Sr	Potassium	K
Chromium	Cr	Nickel	Ni	Calcium	Ca	Sodium	Na
		Cobalt	Co			Magnesium	Mg

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TRYING AND ENTERING THE ANALYTICAL REACTIONS.

204. In order to become fully acquainted with the behaviour of different substances with *reagents*, and thus to be able to detect them by their *reactions*, each of these reactions should be tried with the substance itself or with one of its compounds.

The appearances which are presented during each of these reactions or tests are carefully observed. The substance will then be easily recognised in a body of unknown composition by means of the test.

As soon as a reaction has been satisfactorily performed, it should be briefly described in the Note-book in a neat and concise manner.

The order of the metal groups is reversed in trying through the reactions, because the reactions for the later groups are more simple than those for the earlier ones.

After the reactions of each group of metals have been tried the student should proceed to make a few analyses of substances containing one metal of that group only, and then of substances containing two or more metals of the group. Directions for making these analyses will be found at the end of the reactions for each group.

205. Abbreviations.—For convenience in hasty writing, it is usual to employ abbreviated formulæ for the compound radicles. These abbreviations are especially convenient for formulæ which are constantly used and for those which are of unusual complexity. They are only employed in the text when they save the use of brackets or of numbers, as when $(NH_4)_2$ is written Am_2.

The following abbreviations will be used :—

Am for (NH_4), the metallic-radicle ammonium.
T ,, $(C_4H_4O_6)$, the acid-radicle of tartrates.
$\bar{A}$,, $(C_2H_3O_2)$, the acid-radicle of acetates.
Cy ,, (CN), the acid-radicle of cyanides.

The formulæ Al_2Cl_6, Fe_2Cl_6, Cr_2Cl_6, $Al_2H_6O_6$, $Fe_2H_6O_6$, and $Cr_2H_6O_6$ are now frequently written $AlCl_3$, $FeCl_3$, $CrCl_3$, AlH_3O_3, FeH_3O_3, and CrH_3O_3. Since these smaller formulæ are supported by theoretical considerations, and cannot be looked upon as merely simplifications, they are used in this book.

The following contracted words are also used :—

expt.	for	experiment.	*insol.*	for	insoluble.
pp.	,,	precipitate.	*sol^n.*	,,	solution.
pp^n.	,,	precipitation.	*cryst^{ne}.*	,,	crystalline.
pp^d.	,,	precipitated.	*col^n.*	,,	coloration.
sol.	,,	soluble.			

206. Entry of Analytical Reactions in the Note-book.—The following example, selected from paragraphs 210–213, will serve as a specimen of the brief entry of reactions in the note-book. The formula of a precipitate may be distinguished in the equations by being overlined.

POTASSIUM (K).—Used KCl.

210. *$PtCl_4$*: stirred on watch-glass: yellow cryst^{ne} pp., sol. in much water and in KHO, insol. in alcohol: $2KCl + PtCl_4 = \overline{K_2PtCl_6}$.

211. *$NaH\overline{T}$*: well shaken in a test-tube: white cryst^{ne} pp., sol. in water, in KHO, and in HCl: $KCl + NaH\overline{T} = \overline{KH\overline{T}} + NaCl$.

212. *Flame col^n.*: pale lilac, crimson through the indigo-prism.

213. *Heated in tube or on platinum foil*: solid KCl melted, but gave no fumes: hence it is not volatile.

GENERAL RULES FOR WORKING.

207. The student should attend carefully to the following precautions. They will materially aid the progress and accuracy of his work. If they are observed from the commencement, they will not be felt irksome.

1. Before commencing work, look through the reagent-bottles belonging to the working-bench. Replace any which are absent, and see that the bottles are arranged in a con-

venient order. Then proceed to fill those which are empty, and to re-label any with imperfect or loose labels.

2. Arrange the requisite apparatus on the back part of the bench, so as to leave the front part free to work upon.

3. Then cleanse all soiled glass and porcelain which is not in use. This ought, however, to be unnecessary, since apparatus should never be put away dirty.

4. Every *liquid reagent* must be perfectly clear. Filter it, if it is not quite clear after it has been shaken.

Then take the reagent-bottle in the right hand, and remove the stopper by grasping it between the fourth finger and the palm of the left hand; hold the stopper in this way while the bottle is being used, and then replace it immediately.

Pour the reagent out of the side of the bottle opposite to that which bears the label, and prevent the last drop from running down the outside by touching the lip of the bottle either with the stopper or against the clean edge of the vessel.

Take a *solid reagent* out of the bottle on a clean knife-blade or spatula, or with a slip of clean card-board or paper.

Re-stopper each bottle and replace it on the shelf in its proper place, with the label outwards, immediately after it has been used. Bottles must never be left standing on the working-bench.

5. If a solution or precipitate has to be put by for some time, always label it in such a way as to recall to mind what the solution or precipitate consists of. **Never trust to memory in this matter.**

6. Do not use brass crucible-tongs for holding hot evaporating-basins containing acids, as some of the brass is apt to be dissolved and introduced into the solution. Move a hot dish either by holding it with a cloth, or by lifting the stand upon which the dish is placed.

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7. **Liquids only** are to be poured down the sinks. All **solid** refuse, such as soiled filters, broken glass, and pieces of charcoal, should be thrown into a special receptacle placed in an accessible position.

8. When a substance is being examined in order to find out its composition, write down in the note-book each test or process, with the result obtained, **as soon as it is completed. Never leave the entry of the results until the analysis is completed.**

This rule cannot be too strongly enforced, as neglect of it always causes uncertainty and confusion. The most convenient form of entry is that of the Analytical Tables.

9. **Cleanliness and neatness** cannot be too carefully attended to. More confusion and error may be caused by using dirty test-tubes, beakers, dishes, and funnels than would be thought possible by the beginner. Work should also be rendered as **systematic** as possible, in order to economise time and thought.

208. The following Directions for Trying the Analytical Reactions should be attended to:—

1. Always use a *solution* of the substance, the reactions of which are to be tried, unless it is specially stated that the *solid* is required. This solution is often called **the 'Original Solution.'**

2. Take about twenty cubic centimetres of the solution in a test-tube or small beaker. If the solid substance is also required, take some of it upon a watch-glass and reduce it to a fine powder.

3. Pour a little of the solution into a perfectly clean test-tube and add a few drops of the reagent to it. More of the reagent may be gradually added afterwards, if necessary.

In trying each of the reactions a separate portion of the original solution is to be taken.

4. Before trying a reaction, read carefully through **the whole of the description** of the results to be obtained, and never pass on without being satisfied that the statements of the book are true, and that the test could be repeated successfully.

5. The water which is used for the tests and reactions must always be **distilled water.** The acids are to be used in a dilute condition, unless it is stated that the strong acid is to be employed. If no special statement to the contrary is found, a reagent is to be used in solution. In all cases KHO and NaHO may be used indifferently the one for the other.

REACTIONS FOR THE METALS.

Reactions which are printed in small type need not be tried if time is limited, but they should be carefully read through.

For the convenience of beginners who are not familiar with chemical formulæ, the chemical name as well as the chemical formula of every substance is given when it is first mentioned. Further assistance will be afforded to the beginner if each label in the laboratory carries both the name and the formula of the substance which it denotes.

A list of chemical names with their corresponding formulæ is also printed at the end of the book (548).

GROUP V.—THE POTASSIUM GROUP.

209. This Group includes K, NH_4, Na, Mg. The first three metals belong to the group of the *alkali metals.*

The members of this Group are not precipitated by any Group-reagent.

POTASSIUM (K).—Use Potassium chloride, KCl.

210. Platinum Chloride ($PtCl_4$), if it is added to some KCl solution on a watch-glass and stirred gently but persistently with a glass rod, gives a yellow crystalline precipitate (K_2PtCl_6), which forms first along the lines where the rod has rubbed the glass.

Stir up the precipitate, and pour off the liquid with the precipitate into three test-tubes. To one part add much distilled water and heat, the precipitate will dissolve. To another part add alcohol, the precipitate will not dissolve. Hence this precipitate is soluble in water, and insoluble in alcohol. Warm the third portion with solution of potassium hydrate (KHO), and show that the precipitate is soluble in that liquid.

Since this precipitate is soluble in caustic alkali, a liquid to which the test has to be applied should always be made neutral or faintly acid (Expt. 227, p. 154) by the addition of dilute hydrochloric acid (HCl) before $PtCl_4$ is added to it.

Since the precipitate is less soluble in alcohol than in water, the addition of alcohol hastens the formation of the precipitate in dilute solutions.

A very dilute solution must be evaporated nearly to dryness and cooled before the test is applied.

211. **Sodium hydrogen tartrate** (NaHT), if it is added to some of the KCl solution in a test-tube, gives a white crystalline precipitate (KHT). The precipitate usually appears only when the mouth of the test-tube is closed with the thumb and the tube is well shaken for some time.

Shake up the precipitate and pour it off with the liquid into four test-tubes. Add to three different portions, water, KHO, and hydrochloric acid (HCl) respectively; the precipitate will dissolve when it is shaken or warmed with each of these liquids. To the fourth portion add alcohol; the precipitate will not dissolve. These experiments indicate the conditions which promote or prevent precipitation, as is explained in the preceding paragraph.

212. **Flame Coloration** (186).—Dip a loop of platinum-wire, which gives no colour to the flame, into some strong KCl solution; or moisten the wire and dip it into some finely powdered KCl. Then hold the loop in the Bunsen-flame. A **pale lilac** coloration will be seen if the KCl is pure. But whatever colour is produced, the flame will always appear **deep crimson red** through the thicker parts of an indigo-prism.

213. **Heat a little Solid KCl** in a small dry test-tube; or powder it finely and heat it on a piece of platinum-foil. The KCl will melt, but it will not pass off as vapour.

AMMONIUM (NH_4).—Use Ammonium chloride, NH_4Cl (or AmCl).

214. **KHO**, if it is poured upon some NH_4Cl either in solution or in the solid state, and heated, causes ammonia gas to be evolved,

$$NH_4Cl + KHO = NH_3 + KCl + H_2O.$$

The ammonia gas is recognised:—

a. By its pungent smell, which is that of common 'smelling-salts.'

b. By turning moistened turmeric-paper brown. The test-paper must be wetted with distilled water, placed upon the end of a glass rod, and held in the test-tube, great care being taken not to bring it into contact with the liquid, or with the sides of the tube.

215. $PtCl_4$, when it is stirred on a watch-glass with some AmCl solution, gives a yellow crystalline precipitate (Am_2PtCl_6). This precipitate is soluble in water and in KHO, but is insoluble in alcohol (**210**).

216. NaHT, when it is shaken for some time in a test-tube with AmCl solution, gives a white crystalline precipitate (AmHT). This precipitate is soluble in water, in KHO, and in HCl, but is insoluble in alcohol (**211**).

217. Flame Coloration.—Pure AmCl gives no flame coloration. But ammonium salts almost invariably impart a yellow colour to the flame, since they usually contain a trace of sodium (**219**).

218. Heat a little Solid AmCl in a dry test-tube. The solid will be completely volatilised, and will be condensed as a white coating or *sublimate* on the upper part of the tube.

SODIUM (Na).—Use Sodium chloride, NaCl.

219. Flame Coloration.—NaCl gives an **intense yellow** colour to the flame. The colour is either invisible, or appears only pale blue, when it is viewed through the indigo-prism, **and never shows the slightest tinge of red** when it is seen through the thicker parts of the prism.

The yellow coloration which it imparts to the flame is the most delicate and trustworthy, and often the only possible, test for sodium.

If both potassium and sodium are present, the yellow coloration of sodium is visible, but the potassium coloration is usually unseen. When the flame is examined through the indigo-prism, however, the crimson coloration due to potassium is at once seen. Prove this by mixing some solutions of KCl and NaCl and examining the flame coloration, which is given by this liquid, both with and without the indigo-prism.

220. Heat a little Solid NaCl in a dry test-tube or on platinum-foil. It will melt without producing white fumes, showing that NaCl is not volatile at a red heat.

MAGNESIUM (Mg).—Use Magnesium sulphate, $MgSO_4.7H_2O$.

The $7H_2O$ represents the *water of crystallisation* (68), which is always present in the solid state in the crystallised salt. Water of crystallisation is entered in this way throughout the book.

221. Sodium hydrogen phosphate (Na_2HPO_4), added after AmCl and ammonium hydrate (AmHO), gives a white crystalline precipitate ($MgAmPO_4.6H_2O$).

$$MgSO_4+AmHO+Na_2HPO_4=MgAmPO_4+Na_2SO_4+H_2O.$$

If the solution is very dilute, the precipitate usually forms only after some time, but its formation is hastened if the liquid is warmed and is then well stirred or shaken. The precipitate is soluble in HCl and in other acids, but is insoluble in AmHO.

222. KHO gives a white precipitate (MgH_2O_2), which is readily soluble in an acid.

223. Ammonium hydrate (AmHO) gives a white precipitate (MgH_2O_2); but if AmCl is added before the AmHO, no precipitate is produced.

224. Ammonium carbonate (Am_2CO_3) only gives a white precipitate ($MgCO_3$) in strong solutions and after standing for some time. AmCl prevents the formation of this precipitate.

225. Flame Coloration.—Pure $MgSO_4$ gives no colour to the flame.

226. Blowpipe-test.—If a little solid $MgSO_4.7H_2O$ is placed in a small cavity on a piece of wood-charcoal, and is then heated in the tip of the outer blowpipe-flame (184), it shines brightly and gives a white unmelted mass of MgO. If this white mass is allowed to cool, and is moistened with several drops of cobalt nitrate solution, and is then again heated to whiteness for some time in the outer blowpipe-flame, it assumes a *delicate pink colour.* This colour is very pale, and is most easily seen when the solid mass is placed upon white paper or porcelain.

DETECTION OF METALS IN GROUP V.

227. After the student has carefully worked through the reactions for the metals in the Fifth Group, he should proceed to apply the experience which he has acquired to the detection of the metals belonging to this Group.

For this purpose several unknown salts and mixtures of salts of these metals should be successively supplied to him, and he should proceed to subject separate portions of the liquid, or of the solution of the solid in a little water, to the tests given in the following Table (**229**).

228. Explanation of the Table.—A glance at this Table will show that tests have been selected which serve to distinguish the metals of this Group when they are present either singly or several together. The order in which these tests are placed will also be seen to be that which most readily effects this object.

When more than one metal of the Fifth Group is present in the substance to be examined, the possibility of interference in the reactions arises. It should be remembered, however, that—

1. Mg is indicated with certainty by its precipitation by Na_2HPO_4 in the presence of AmCl and AmHO, and by its precipitation by KHO solution.

2. Na can always be found by its yellow flame coloration ; and K may always be found by a crimson flame coloration being seen through the indigo-prism.

3. NH_4 may always be detected by the evolution of NH_3 when the substance is boiled with KHO solution.

4. The test for K by means of $PtCl_4$ or NaHT is only conclusive in the absence of NH_4. If NH_4 is present, therefore, its salts must be removed by ignition before K can be tested for by precipitation.

These statements will serve to explain the correspondingly numbered tests in the Table.

It will generally be unnecessary to try the somewhat complicated confirmatory test (IV) for K, since K may be detected with certainty by its flame coloration.

In future tables the bracketed paragraphs, which concern the absence of the metals, will be omitted, but the student should invariably enter his negative results, and the inferences which he draws from them.

Sodium is rarely entirely absent from any substance. The quantity of this metal which is present may be judged from the intensity of the flame coloration. Unless the colour is intense, only a trace of sodium may be inferred to be present.

An example of the form of entry of the results of an analysis is given in paragraph 230.

229. TABLE FOR TESTING FOR Mg, K, Na, NH_4, IN A SOLUTION WHICH MAY CONTAIN ALL OF THEM.

Experiment.	Observation.	Inference.
I. Add to a small part of the solution AmCl, then AmHO and Na_2HPO_4, shake well and allow to stand if no precipitate appears at once.	1. A white precipitate forms. [2. No precipitate appears.]	Presence of Mg. [Absence of Mg.]
II. Dip a small loop of clean platinum wire into the solution, and hold it near the top of the Bunsen-flame. Examine the flame coloration through the indigo-prism.	1. A yellow flame coloration. A crimson red colour is seen through the indigo-prism. 2. A pale violet flame, appearing crimson red when seen through the indigo-prism. [3. No colour is imparted to the flame.]	Presence of Na, possibly also of K. Presence of K. Presence of K and absence of Na. [Absence of K and Na.]
III. Boil some of the solution in a test-tube with KHO solution, and try whether NH_3 is given off by smelling; or if it cannot be smelt, by testing with moistened turmeric paper.	1. NH_3 gas is smelt, and the turmeric-paper becomes brown. [2. No NH_3 is smelt, and the turmeric-paper does not become brown.]	Presence of NH_4. [Absence of NH_4.]
IV. A confirmatory test for K may be made by adding $PtCl_4$ or NaHT. If Expt. III. has shown that NH_4 is present, proceed to test for K by column A (page 169); if NH_4 is absent, test for K by column B.		

A.	B.
Boil some more of the solution down to dryness in a porcelain-dish; scrape out the white substance left in the dish and heat it strongly on a piece of platinum-foil as long as any white fumes are seen to be given off when the foil is taken out of the flame for an instant. Place the foil when cold in a test-tube, and boil it with *very little* water, to which a drop of dilute HCl has been added, and evaporate down to several drops; cool the liquid, and pour it out upon a watch-glass, add a little $PtCl_4$, and stir well:— A yellow precipitate:—*Presence of K.*	Pour a little of the solution upon a watch-glass, add $PtCl_4$, and stir well: a yellow precipitate:—*Presence of K.* If no precipitate forms, K may nevertheless be present, because the solution is too dilute to allow the precipitate to form; therefore pour some of the solution into a porcelain-dish, boil down nearly to dryness, cool and test this liquid by stirring it with $PtCl_4$ on a watch-glass, adding alcohol if the precipitate does not appear at once: a yellow precipitate:—*Presence of K.* *Note.*—Addition of alcohol will frequently produce the precipitate without evaporation.

230. **Example of Entry.**—As each test is made, the result which it yields should be carefully observed; a description of the experiment, followed by the observation and the inference, should be at once carefully written down in the note-book.

The following example will serve to illustrate the method of entering the results of an analysis.

A solid white substance was given to be tested for the Metals in Group V.

The substance was dissolved by warming it with a little water.

Test.	Observation.	Inference.
1. Added AmCl, AmHO and Na_2HPO_4.	No precipitate.	Absence of Mg.
2. Dipped a platinum wire loop in the solution and held it in the flame.	A yellow coloration. The flame appeared crimson through the indigo-prism.	Presence of Na. Presence of K.
3. Boiled the solution with KHO solution.	No smell of NH_3.	Absence of NH_4.
4. Stirred with $PtCl_4$.	Yellow precipitate.	Presence of K confirmed.
		Found Na and K.

GROUP IV.—BARIUM GROUP.

231. This Group includes Ba, Sr, Ca.

These metals differ from those of Group V. by being precipitated as carbonates by ammonium carbonate (Am_2CO_3), and as oxalates by ammonium oxalate ($Am_2C_2O_4$), in the presence of ammonium chloride (AmCl). They are not precipitated by any other Group-reagent. The carbonates are soluble in boiling solution of AmCl.

BARIUM (Ba).—Use Barium chloride, $BaCl_2.2H_2O$.

232. Ammonium carbonate (Am_2CO_3), added after ammonium chloride (AmCl), produces a white precipitate ($BaCO_3$), which is soluble in acetic acid ($H\bar{A}$).

233. Calcium sulphate ($CaSO_4$), or Sulphuric acid (H_2SO_4), causes a heavy white precipitate ($BaSO_4$), which is insoluble in hydrochloric acid (HCl) even on boiling.

234. Potassium chromate (K_2CrO_4), added after some acetic acid ($H\bar{A}$), produces a yellow precipitate ($BaCrO_4$), which is soluble in warm HCl, but differs from $SrCrO_4$ by being insoluble in warm $H\bar{A}$.

235. Flame Coloration: yellowish-green, visible through the indigo-prism.

STRONTIUM (Sr).—Use Strontium nitrate, $Sr(NO_3)_2.4H_2O$.

236. Am_2CO_3, added after some AmCl, gives a white precipitate, strontium carbonate ($SrCO_3$), which is soluble in $H\bar{A}$.

237. $CaSO_4$, or H_2SO_4, gives a white precipitate ($SrSO_4$).

This precipitate does not usually form in a cold solution at once, but only after some time. The precipitate, however, **appears at once when the liquid is boiled.**

238. **K_2CrO_4**, added after some $H\bar{A}$, causes no precipitate, since $SrCrO_4$ is soluble in $H\bar{A}$.

239. Flame Coloration: crimson-red, appearing *intense red* through the indigo-prism.

CALCIUM (Ca).—Use Calcium chloride, $CaCl_2.6H_2O$.

240. Am_2CO_3 added after AmCl: white precipitate ($CaCO_3$), soluble in $H\bar{A}$.

241. $CaSO_4$: no precipitate, even when the liquid is allowed to stand, or is boiled.

242. H_2SO_4: a white precipitate ($CaSO_4$) forms at once in strong solutions, and often in weak solutions when they are boiled; but some $CaSO_4$ always remains in solution, since it is not quite insoluble in water.

Prove this by adding more H_2SO_4 until its addition produces no further precipitate. Then boil the liquid containing the precipitate, and filter it. Add AmHO in excess (**191**) to the clear filtrate, and a little $Am_2C_2O_4$; a white precipitate (CaC_2O_4) will form, showing the presence of Ca, which must have been dissolved as $CaSO_4$.

243. K_2CrO_4 gives no precipitate.

244. Ammonium oxalate ($Am_2C_2O_4$): white precipitate (CaC_2O_4), soluble in most acids, but insoluble in $H\bar{A}$.

A similar precipitate is produced by $Am_2C_2O_4$ in solutions of barium and strontium salts.

245. **Flame Coloration**: yellowish-red. When this coloration is viewed through the indigo-prism, it *does not appear red*, and differs in this respect from that given by Sr.

DETECTION AND SEPARATION OF METALS IN GROUP IV.

246. **The Following Differences** in the reactions of salts of Ba, Sr, and Ca are made use of in drawing up the Table in paragraph 517.

1. The difference in the flame colorations yielded by these metals, which are examined if necessary through the indigo-prism.

2. The difference in the behaviour of their solutions with $CaSO_4$ solution, which at once indicates the presence or absence of Ba, or of Sr in the absence of Ba.

3. The insolubility of $BaCrO_4$ and solubility of $SrCrO_4$ and $CaCrO_4$ in $H\bar{A}$, and hence the possibility of separating Ba, if it is present, by adding $H\bar{A}$ and then K_2CrO_4.

4. The complete precipitation of Sr, if it is present, by boiling the solution with H_2SO_4; sufficient $CaSO_4$ still remaining in solution to give a precipitate of CaC_2O_4 on the addition of $Am_2C_2O_4$ to the filtrate, after it has been made alkaline with AmHO.

247. **A Solution which has to be examined for Ba, Sr, and Ca** is first made alkaline by the addition of AmHO.

Am_2CO_3 is then added in excess (see *Note* below).

The precipitate, which contains all the metals of Group IV. originally present in the solution, is then examined by Table IV. (517).

Note.—The addition of the reagent is continued as long as it causes any further precipitate, after the liquid has been stirred well and allowed to settle. The liquid is then filtered. A little more Am_2CO_3 is added to the clear filtrate, and if this causes any further precipitate more Am_2CO_3 is added, and the liquid is again poured through the same filter. This process is repeated until the clear filtrate gives no precipitate with Am_2CO_3.

GROUP III.A.—IRON GROUP.

248. This Group includes Al, Fe, Cr.

The members of this Group differ from those of Groups III.B, IV., and V. by being precipitated by AmHO added after AmCl. They are not precipitated, however, by the Group-reagents for Groups II. and I.

This Group is also precipitated by ammonium sulphide (Am_2S), and by hydrogen sulphide (H_2S) when it is added in excess to the alkaline solution.

ALUMINIUM (Al).—Use Ammonium alum, $AlAm(SO_4)_2.12H_2O$.

249. Ammonium hydrate (AmHO): white flocculent precipitate (AlH_3O_3),* soluble in hydrochloric acid (HCl) and in acetic acid (HA), and slightly soluble in AmHO in the absence of ammonium chloride (AmCl):—

$$AlAm(SO_4)_2 + 3AmHO = AlH_3O_3 + 2Am_2SO_4.$$

250. Potassium hydrate (KHO), added in very small quantity: white flocculent precipitate (AlH_3O_3), soluble in excess of KHO.

If to a part of the solution in KHO, HCl is added gradually until the liquid is neutral, AlH_3O_3 is reprecipitated. If more acid is added the precipitate dissolves, but it reappears when AmHO is added in slight excess.

From another portion of the solution in KHO the AlH_3O_3 may be reprecipitated by the addition of sufficient AmCl solution.

251. Ammonium sulphide (Am_2S) yields the same precipitate (AlH_3O_3), H_2S gas being either evolved or remaining dissolved in the liquid:—

$$2AlAm(SO_4)_2 + 3Am_2S + 6H_2O = 2AlH_3O_3 + 4Am_2SO_4 + 3H_2S$$

---

* This formula is sometimes written $Al_2H_6O_6$.

252. Blowpipe-test.—Heat some solid $AlAm(SO_4)_2.12H_2O$ on charcoal in the outer blowpipe-flame; then moisten it, after cooling, with $Co(NO_3)_2$ solution and again heat it in the outer flame; a *fine blue mass* will be obtained.

IRON (Fe).—Use Ferric chloride ($FeCl_3$), and Ferrous sulphate ($FeSO_4.7H_2O$).

Use $FeCl_3$ and $FeSO_4$ solutions for **254**, **255**, **256**, and **259**, and solid $FeSO_4.7H_2O$ for **257** and **258**. Prepare $FeSO_4$ solution by crushing a crystal of the solid substance and shaking it with *cold* water in a test-tube just before the solution is used.

253. Two classes of iron-compounds are known which differ in appearance and properties and behave differently with reagents. They are known as *ferrous* and *ferric* compounds respectively. Ferric salts are usually yellow or reddish-yellow. Ferrous salts are usually pale green if they are in solution, or are combined with water (hydrated), and white if they are perfectly free from water (anhydrous).

Ferrous compounds are readily converted into ferric compounds by boiling them for a short time with a little strong HNO_3, or with HCl and a crystal of $KClO_3$.

Both classes of iron-compounds give the same results in the blowpipe reactions (**257**, **258**). Differences are noticeable, however, in their other reactions.

254. AmHO, or KHO: with *ferric salt*, a reddish-brown flocculent precipitate (FeH_3O_3), insoluble in excess, soluble in HCl: with *ferrous salt*, a dingy-green precipitate, which turns brown by oxidation when it is exposed to the air.

255. Am_2S: a black precipitate (FeS), which is mixed with white sulphur in the case of *ferric salt*:—$2FeCl_3 + 3Am_2S = \overline{2FeS + S} + 6AmCl$. FeS is soluble in boiling acids, but insoluble in KHO.

In a very dilute iron-solution a green colour only is produced at first by Am_2S, but black FeS separates after the liquid has stood for some time.

256. Hydrogen sulphide (H_2S), passed as gas into the solution, or added as H_2S-water: black precipitate (FeS) in alkaline solutions.

In neutral or acid solution of *ferric salt*, sulphur is precipitated and the ferric salt is changed into a ferrous salt; the colour of the solution therefore changes from yellow to pale green, as is seen when the strong solution has been boiled and filtered:—$2FeCl_3 + H_2S = 2FeCl_2 + 2HCl + S$.

257. **Heating with Fused Sodium carbonate (Na_2CO_3) and Potassium cyanide (KCy) on Charcoal in the inner blowpipe-flame** separates a grey magnetic powder (184) from a solid iron-compound. The residue on the charcoal is washed by levigation (185), and the metallic powder is then shown to be attracted when it is touched under water with the point of a magnetised knife-blade.

258. **A Borax-bead (183)** containing Fe is *reddish-brown* while hot, and *yellow* when cold, after it has been fused in the outer blowpipe-flame: after it has been fused in the inner flame it is *olive green.*

259. **Distinctive tests for Ferrous and Ferric salts.**—For these tests several drops of dilute HCl should be first added to the iron solution. Ferrous solutions usually contain some ferric salt.

Reagents to be added.	Ferric salt. (Use $FeCl_3$ solution.)	Ferrous salts. (Use $FeSO_4$ solution.)
1. **Potassium ferrocyanide (K_4FeCy_6)** added in small quantity:	*Dark-blue precipitate,* 'Prussian blue'; turned brown by KHO.	*Light-blue precipitate.*
2. **Potassium ferricyanide (K_3FeCy_6):**	*No precipitate*: the solution darkens.	*Dark-blue precipitate,* 'Turnbull's blue.'
3. **Potassium sulphocyanide (KCyS):**	*Blood-red coloration.*	*No red coloration.*
4. **Potassium hydrate (KHO):**	*Reddish-brown* precipitate.	*Dingy-green* precipitate, turning brown in the air.

CHROMIUM (Cr).—Use Chrome alum, $CrK(SO_4)_2.12H_2O$.

Chromic salts are usually bluish-green or violet in colour.

260. **AmHO**: pale bluish-green or purple precipitate (CrH_3O_3).

If AmHO is added in large quantity and the liquid is heated, some of the precipitate dissolves, producing a beautiful violet-red solution. The colour of the liquid is best seen after the precipitate has been removed by filtration. CrH_3O_3 is precipitated from this liquid when it is boiled for several minutes in a porcelain-dish.

261. **KHO**, if it is added in small quantity, gives the same precipitate (CrH_3O_3). If more *cold* KHO is added, the precipitate is entirely dissolved to a green fluid. When this liquid is diluted with water and is boiled for several minutes, CrH_3O_3 is reprecipitated. This may also be effected by adding AmCl.

262. **Am_2S** also precipitates CrH_3O_3, H_2S gas being given off or remaining dissolved, as in the reaction in paragraph 251.

263. **When a Solid substance containing Cr is heated with fused Na_2CO_3 and potassium nitrate (KNO_3)** on platinum-foil it yields a yellow mass, containing yellow Na_2CrO_4, and if this is dissolved in boiling water a yellow solution is obtained. If the solution is acidified with acetic acid ($H\bar{A}$) and boiled for a few minutes to drive off carbon dioxide (CO_2), and is then mixed with lead acetate ($Pb\bar{A}_2$) solution, a yellow precipitate ($PbCrO_4$) is obtained.

264. **A Borax-bead** containing Cr is green after it has been fused in the outer or the inner blowpipe-flame.

DETECTION AND SEPARATION OF METALS IN GROUP III.A.

265. The following Differences and Tests are made use of in drawing out the Table in paragraph 514.

1. The solubility of AlH_3O_3 in *boiling* KHO, in which FeH_3O_3 and CrH_3O_3 are insoluble.

2. The conversion of CrH_3O_3, when it is heated with fused Na_2CO_3 and KNO_3, into soluble Na_2CrO_4 : FeH_3O_3 when thus treated remaining as Fe_2O_3, which is insoluble in water.

3. The detection of Cr by the yellow colour of the solution of the Na_2CrO_4 (2) in water, and by the formation of a yellow precipitate when the solution is acidified with $H\bar{A}$ and $Pb\bar{A}_2$ is added.

4. The detection of Fe by dissolving the Fe_2O_3 (2) in HCl, and obtaining a blood-red coloration when KCyS is added to the solution.

266. A Solution which may contain Al, Fe, and Cr is examined in the following manner.

I. A small part of the solution is acidified with HCl. A little K_4FeCy_6 is added to a few drops of this acidified solution. If no blue precipitate forms, Fe is absent. If a blue precipitate is produced, Fe is present, and the following further tests for ferrous and ferric iron must be made.

Another portion of the acidified solution is tested with freshly-made K_3FeCy_6 solution, a dark-blue precipitate shows the presence of Fe'' (ferrous salt).

A third portion of the solution is tested with KCyS, which may produce a blood-red coloration, proving the presence of Fe''' (ferric salt).

II. Boil the rest of the original solution, after adding a few drops of strong HNO_3 if a ferrous salt is present. Add some AmCl and then AmHO gradually to the liquid, until it is just alkaline and smells faintly of NH_3 after it has been well stirred. Now boil the liquid, filter off the precipitate, and examine it by Table III.A (514), disregarding all the appended notes.

GROUP III.B.—ZINC GROUP.

267. This Group includes Zn, Mn, Ni, Co.

Compounds of these metals differ from those of the metals of Groups IV. and V. by being precipitated from their neutral or alkaline solutions by Am_2S, and from their alkaline solutions by H_2S. They are not precipitated by the Group Reagents for Groups III.A., II., and I. Mn is, however, gradually precipitated by oxidation, when its solution is mixed with AmCl and excess of AmHO, and is then exposed to the air.

ZINC (Zn).—Use Zinc sulphate, $ZnSO_4.7H_2O$.

Zinc salts are usually colourless.

268. Ammonium sulphide (Am_2S): white precipitate (ZnS). This precipitate often appears to be yellow, if a large excess of yellow Am_2S has been added. It is soluble in HCl and insoluble in $H\bar{A}$ and in Am_2S.

NOTE.—Unless freshly prepared *colourless* Am_2S can be obtained, the tests which show the solubility of the sulphides of the metals of this Group should be tried on the precipitate which is obtained by the addition of AmCl, AmHO in excess, and H_2S; since the *yellow* ammonium polysulphide solution causes white sulphur to precipitate,

$$ZnSO_4 + Am_2S_2 = ZnS + S + Am_2SO_4,$$

and this substance is usually insoluble in the liquids which dissolve the precipitated metallic sulphide.

269. Hydrogen sulphide (H_2S): white precipitate (ZnS). A neutral solution of Zn is only partially precipitated by H_2S, since H_2SO_4 is formed by the reaction and arrests the action of H_2S :—$ZnSO_4 + H_2S = ZnS + H_2SO_4$. But if ammonium hydrate (AmHO) has been previously added in excess, the Zn may be entirely precipitated by H_2S. The addition of sufficient sodium acetate ($Na\bar{A}$) solution also ensures complete precipitation, since the H_2SO_4 in the solution is thus replaced by $H\bar{A}$, in which ZnS is insoluble :—

$$ZnSO_4 + H_2S + 2Na\bar{A} = ZnS + Na_2SO_4 + 2H\bar{A}.$$

270. **Potassium hydrate (KHO)** gives a white precipitate (ZnH_2O_2) which is soluble in excess. The zinc may be precipitated as ZnS from this potassium hydrate solution by the addition of H_2S.

271. **Blowpipe - test.** — Mix some finely - powdered $ZnSO_4.7H_2O$ with solid sodium carbonate (Na_2CO_3) on charcoal, and heat the mixture in the inner blowpipe-flame. An incrustation is obtained, which is *yellow* while hot and *white* when cold.

Moisten this incrustation with cobalt nitrate solution and heat it again strongly in the outer blowpipe-flame, it will become *green*.

MANGANESE (Mn).—Use Manganese chloride ($MnCl_2$) or Manganese sulphate ($MnSO_4$).

Manganous salts are usually *pale-pink* in colour.

272. **Am_2S** : flesh-coloured or pale pink precipitate (MnS): the colour of the precipitate may appear yellow if excess of yellow Am_2S has been added: its true colour and its solubility may be observed by proceeding as is directed in the Note in paragraph 268. The colour of the moist filtered precipitate changes by oxidation to dark brown when it is allowed to stand for some time exposed to the air (274).

273. H_2S precipitates MnS partly from neutral solutions (269), entirely from alkaline solutions, but not at all in presence of free hydrochloric acid (HCl) or acetic acid ($H\bar{A}$). Add to some $MnCl_2$ solution ammonium chloride (AmCl), ammonium hydrate (AmHO) in excess, and then H_2S ; and show with separate portions of the precipitate thus obtained that MnS is soluble in HCl and in $H\bar{A}$, but is insoluble in Am_2S.

274. **KHO**: white precipitate (MnH_2O_2), insoluble in excess, and quickly turning brown by oxidation when it is exposed to the air. This change in colour is most rapid

when the precipitate is separated from the liquid by filtration.

275. **Ammonium hydrate (AmHO)** gives the same precipitate as KHO does, but if sufficient AmCl has been first added, AmHO produces no immediate precipitate. This ammoniacal solution, however, gradually turns brown by oxidation when it is exposed to the air, and the Mn is ultimately precipitated as a brown hydroxide.

276. **Blowpipe-test.**—When any solid substance containing Mn is heated with a fused mixture of Na_2CO_3 and a little potassium nitrate (KNO_3) *upon platinum-foil*, it yields a *bluish-green* mass. This reaction is very delicate.

277. **A Borax-bead** containing *very little* Mn is *violet-red* while it is hot and *amethyst-red* when it is cold, after it has been fused in the outer blowpipe-flame. After the bead has been fused for some time in the inner flame it becomes *colourless.*

NICKEL (Ni).—Use Nickel sulphate, $NiSO_4.7H_2O$.

Hydrated nickel salts are usually *bright green* in colour: the anhydrous salts are yellow.

278. **Am_2S** : black precipitate (NiS), which is insoluble in cold dilute HCl and in $H\bar{A}$ (279), but is dissolved by being heated with HCl and a crystal of potassium chlorate ($KClO_3$).

The precipitate is slightly soluble in *yellow* Am_2S. Prove this by adding more *yellow* Am_2S, and then boiling and filtering the liquid. The filtrate will be coloured brown by NiS dissolved in the excess of Am_2S. Boil this filtrate in a porcelain-dish for some time, adding water if necessary. The dissolved NiS will be reprecipitated, and when this has been filtered off, a colourless filtrate will remain.

279. **H_2S** : black precipitate (NiS) in neutral or alkaline solution, or in solution containing no free acid except $H\bar{A}$ (269). Add AmCl, AmHO in excess, and then H_2S, to the $NiSO_4$ solution in order to obtain this precipitate.

280. AmHO, if it is added in very small quantity, gives a bluish-green precipitate (NiH_2O_2). This precipitate is soluble in excess of AmHO to a violet-blue liquid; it is also soluble in AmCl. Accordingly, if AmCl is first added to the $NiSO_4$ solution, the addition of AmHO causes no precipitate.

281. Potassium cyanide (KCy) solution, if it is freshly prepared and is added in small quantity, gives a yellowish-green precipitate ($NiCy_2$). By the further addition of this solution, this precipitate is dissolved; but it is reproduced when sufficient HCl is added to the cyanide solution.

282. Acidify some $NiSO_4$ solution with several drops of $H\bar{A}$. Then add KCy solution gradually, while stirring, until the precipitate which forms at first is just re-dissolved. Boil this solution for a short time. Then cool it, and divide it into two parts.

The addition of HCl in excess to one part will produce a precipitate of $NiCy_2$, which often appears only after a time.

The addition to the other part of excess of KHO, and then of sufficient Br-water to cause the liquid to remain yellow, produces potassium hypobromite (KBrO), and this yields a black precipitate (NiH_8O_8) when the liquid is warmed.

283. A Borax-bead containing Ni is *violet* or *reddish-brown* after it has been fused in the outer blowpipe-flame, and becomes *pale yellow* when it is cold; it is *black* and *opaque* after it has been fused in the inner flame.

COBALT (Co.)—Use Cobalt nitrate, $Co(NO_3)_2.6H_2O$.

Hydrated or dissolved cobalt salts are usually *reddish-pink* in colour; the solid anhydrous salts are usually *blue* or *purple*. Cobalt iodide is green.

284. Am_2S: black precipitate (CoS). When much yellow Am_2S is added, and the liquid is boiled and filtered, the filtrate is coloured *yellow* by the Am_2S; it is *not dark-coloured*, since CoS differs from NiS by being insoluble in yellow Am_2S. The behaviour of CoS with other reagents resembles that of NiS (278, 279).

285. **AmHO**: bluish-green precipitate. The precipitate is soluble in excess of AmHO and in AmCl; it is therefore not formed if AmCl is added before AmHO.

286. **KCy** solution, if it is freshly prepared and is added in small quantity, gives a reddish-brown precipitate ($CoCy_2$). When more KCy solution is added gradually and with constant stirring the precipitate dissolves, but it reappears when HCl is added to the cyanide solution.

287. Acidify some $Co(NO_3)_2$ solution with a few drops of $H\bar{A}$; then add KCy solution gradually until the precipitate, which forms at first, is just re-dissolved. Boil this liquid in an open dish; then cool it and divide it into two parts. Add to one of these portions HCl, and to the other excess of KHO solution and then Br-water till the liquid remains yellow; no precipitates will appear even when these liquids are heated.

This difference of behaviour of Co and Ni solutions (**282**), which have been boiled with excess of KCy, is due to the fact that $NiCy_2$ forms with KCy a compound ($NiCy_2.2KCy$) which is easily decomposed: whereas $CoCy_2$ forms with KCy the very stable compound K_3CoCy_6, *potassium cobalticyanide.*

288. **A Borax-bead** containing Co is *fine blue* in colour after it has been fused in the inner or in the outer blowpipe-flame.

DETECTION AND SEPARATION OF METALS IN GROUP III.B.

289. The processes used in Table III.B. (**516**) depend upon the following differences in the behaviour of the metals of this Group.

1. The solubility of NiS in yellow Am_2S, in which ZnS, MnS, and CoS are insoluble: the precipitation of NiS from this solution by boiling the liquid, and the confirmation of the presence of Ni by fusing the precipitated NiS in a borax-bead.

2. The solubility of ZnS and MnS in cold dilute HCl, in which NiS and CoS are almost insoluble.

3. The solubility of ZnH_2O_2 in cold KHO, in which MnH_2O_2 is insoluble; the precipitation of white ZnS from this solution by H_2S,

and the production of a bluish-green mass by heating the undissolved MnH_2O_2 with fused Na_2CO_3 and KNO_3.

4. The difference of behaviour with KBrO of the KCy solutions of $NiCy_2$ and of $CoCy_2$: and the marked difference in the colour of borax-beads which contain Ni and Co respectively.

290. A Solution is examined for Zn, Mn, Co, and Ni by adding AmCl, then AmHO until it is alkaline, and then yellow Am_2S until the liquid smells of it after it has been well stirred. The liquid is then heated, and a few drops of it are poured upon a filter. If the filtrate is not yellow or brown in colour, more yellow Am_2S must be added until the filtrate becomes coloured. The whole liquid is then heated and filtered, and the precipitate is examined by Table III.B. (516).

If the filtrate is yellow, it is rejected. If it is brown, it is examined for Ni by boiling it until the NiS separates (278), and this NiS is then fused into a borax-bead (283).

GROUP II.A.—COPPER GROUP.

291. This Group includes Hg″, Pb, Bi, Cu, Cd.

The compounds of these metals differ from those of the metals in Groups III.A, III.B, IV., and V. by being precipitated as sulphides by H_2S from their acid solutions. With the exception of Pb, these metals are not precipitated by HCl or by soluble chlorides. A strong solution of Pb is partially precipitated as chloride by these reagents.

The sulphides of these metals differ from those of Group II.B by being insoluble both in Am_2S and in KHO.

If very much acid is present, H_2S does not precipitate the members of this group readily or completely, unless their solutions are largely diluted with water.

MERCURICUM (Hg″).—Use Mercuric chloride, $HgCl_2$.

Mercury forms two series of compounds, which are distinguished as *mercuric* and *mercurous*. The metal in these two series of compounds behaves differently with reagents, and may be distinctively called *mercuricum* and *mercurosum*. In the latter condition it is classed under Group I.

292. Hydrogen sulphide (H_2S): black precipitate (HgS). If H_2S or its solution is added slowly, the precipitate is first *white*, then *brown* or *orange*, and ultimately becomes *black*. The occurrence of these changes of colour during the addition of H_2S is characteristic of mercuric salts.

If the precipitate is filtered off, and is then washed until it is free from dissolved chlorides, it may be proved to be insoluble in hot strong nitric acid (HNO_3) and in ammonium sulphide (Am_2S), but easily soluble in a mixture of HNO_3 and hydrochloric acid (HCl), which is known as *aqua regia*.

293. ~~**Stannous chloride**~~ **($SnCl_2$)**: white precipitate (Hg_2Cl_2): if more $SnCl_2$ is added, the white precipitate becomes grey, and consists of fine particles of Hg.

$$2HgCl_2 + SnCl_2 = Hg_2Cl_2 + SnCl_4$$
$$Hg_2Cl_2 + SnCl_2 = 2Hg + SnCl_4.$$

294. **Copper (Cu)**: cleanse a small strip of copper sheet or a copper coin, by rubbing it with sand-paper, or by dipping it into strong HNO_3. Immerse it in some $HgCl_2$ solution made acid with several drops of HCl. The Cu will become coated with a grey film of Hg:—$HgCl_2 + Cu = Hg + CuCl_2$.

If the surface of the strip is rubbed, it will appear more or less whitened like silver, since the mercury has formed an *amalgam* with the copper; and when the copper strip is heated in a test-tube, a sublimate of globules of Hg will form upon the cool sides of the tube.

295. **AmHO** gives a white precipitate (NH_2HgCl), insoluble in excess.

296. **KHO** gives a yellow precipitate of mercuric oxide (HgO).

297. **Mercury Mirror.**—Mix *a little* finely powdered $HgCl_2$ intimately with about four times as much finely powdered **sodium carbonate** (Na_2CO_3) which has been recently dried by ignition. Pour this mixture into a small *dry* test-tube: then cover the mixture with a layer of dry Na_2CO_3.

After the inside of the tube has been perfectly cleansed, heat the upper layer of Na_2CO_3 strongly; and, while keeping this hot, gradually extend the heat to the mixture. Hg will sublime, condensing in globules on the inside of the tube, and usually appearing as a mirror.

The globules are often only seen by the aid of a lens, or when they are flattened by pressing them with a glass rod against the inside of the tube.

298. **Sublimation.**—Heat a little solid $HgCl_2$ in a tube closed at one end; the substance will sublime in white fumes, showing that mercury compounds are volatile.

LEAD (Pb).—Use Lead acetate, $Pb\bar{A}_2.3H_2O$, dissolved in very dilute $H\bar{A}$.

299. **H_2S**: black precipitate (PbS). This precipitate is often *red*, if much hydrochloric acid is present; but it becomes black when water is added and H_2S is passed, or when H_2S-water is added. Filter off this precipitate, and

show with separate portions that PbS is insoluble in potassium hydrate (KHO) and in ammonium sulphide (Am_2S), but soluble in boiling dilute HNO_3; also that it is converted by boiling strong HNO_3 into white insoluble $PbSO_4$.

300. **Hydrochloric acid (HCl)**: white precipitate ($PbCl_2$), forming only in cold and strong solutions. When this precipitate is boiled with sufficient water, it dissolves; but it separates again in beautiful crystals when the solution is cooled.

301. **Sulphuric acid (H_2SO_4)**: white precipitate ($PbSO_4$). This precipitate is much less soluble in dilute H_2SO_4 than in water; hence H_2SO_4 should be added in excess to a pretty strong solution. Decant the liquid and show that the precipitate is dissolved by boiling it either with $Am\bar{A}$ solution, or with acetic acid $H\bar{A}$ after adding ammonium hydrate (AmHO) in excess; show also that it dissolves in boiling strong HCl.

302. **Potassium chromate (K_2CrO_4)**: yellow precipitate ($PbCrO_4$), soluble in KHO, but insoluble in acetic acid ($H\bar{A}$).

303. **Blowpipe-test.**—Mix some powdered $Pb\bar{A}_2.3H_2O$ with about twice as much Na_2CO_3 in a cavity on a piece of charcoal, and heat the mixture in the inner blowpipe-flame. A yellow incrustation will form around the cavity, and small lustrous globules of Pb will be seen within. Detach and cleanse one of these globules, and prove that it is malleable and will mark paper (184).

BISMUTH (Bi).—Use Bismuth nitrate solution, produced by dissolving $BiO(NO_3).H_2O$ or $Bi(NO_3)_3.5H_2O$ in very dilute HNO_3.

304. **H_2S**: brownish-black precipitate (Bi_2S_3):—

$$2Bi(NO_3)_3 + 3H_2S = Bi_2S_3 + 6HNO_3:$$

insoluble in Am_2S and in KHO, but soluble in strong HNO_3.

305. **Ammonium hydrate (AmHO), or Potassium hydrate (KHO)**: white precipitate (BiH_3O_3), insoluble in excess. Filter off the precipitate and keep it for the following reaction.

306. **Water (H_2O).**—Pour *a few drops* of boiling dilute HCl upon the precipitate which was left in the filter from the last reaction, and allow the acid solution to drop from the funnel into a large quantity of distilled water. A *milkiness* will appear in the water, which is due to the formation of BiOCl:—$BiCl_3 + H_2O = BiOCl + 2HCl$.

A similar precipitate ($BiONO_3$) may be obtained by letting the original bismuth nitrate solution drop into much water.

These precipitates are soluble in HCl, but are insoluble in solution of tartaric acid ($H_2\bar{T}$).

307. **Blowpipe-test.**—Heat a mixture of bismuth salt and Na_2CO_3 and KCy on charcoal in the inner blowpipe-flame. An incrustation will be obtained which is *orange-red* while it is hot and *yellow* when cold; white globules of Bi will also be seen; these are very brittle, and are easily crushed to powder by a sharp blow with the pestle, and they will not mark paper (184).

COPPER (Cu).—Use Copper sulphate, $CuSO_4.5H_2O$.

308. **H_2S**: brownish-black precipitate (CuS). Filter, keeping the funnel carefully covered with a glass plate, since air quickly oxidises CuS to $CuSO_4$, and this dissolves and runs through with the filtrate. Show that the precipitate is insoluble in KHO, in Am_2S, and in boiling dilute H_2SO_4, but dissolves in boiling HNO_3, and in KCy solution.

309. **AmHO**, if it is added in a diluted state and in very small quantity, gives a *greenish blue* precipitate. If more AmHO is added, this precipitate dissolves, yielding an *intensely blue* liquid.

310. **KHO** produces a pale-blue flocculent precipitate, which becomes black when the liquid is heated.

311. **Potassium ferrocyanide** (K_4FeCy_6): reddish-brown precipitate (Cu_2FeCy_6) insoluble in $H\bar{A}$. In a *very dilute* copper solution only a reddish colour is seen at first.

312. **Iron** (**Fe**).—Cleanse a bright piece of steel or iron, such as the blade of a penknife, by rubbing it with sandpaper; then dip it into $CuSO_4$ solution which has been made acid with a few drops of H_2SO_4; the surface of the iron will be gradually covered with a red film of Cu.

313. **Blowpipe-test.**—If a mixture of finely powdered $CuSO_4.5H_2O$ with KCy and Na_2CO_3 is made in a cavity on charcoal and the mixture is heated in the inner blowpipe-flame, red scales of Cu are obtained. These are best seen after they have been separated by levigation (185).

314. **A Borax-bead** containing Cu is *green* while hot and *blue* when cold after it has been fused in the outer blowpipe-flame. After it has been fused in the inner flame the bead becomes *red* or *colourless*, if too much copper is not present; this change is facilitated by fusing a little Sn into the bead.

315. **The Flame coloration** produced by a copper-salt is *green*, and shows a *blue centre* if the copper compound has been moistened with HCl, or if copper chloride ($CuCl_2$) was originally used.

CADMIUM (Cd).—Use Cadmium sulphate, $CdSO_4.4H_2O$.

316. **H_2S**: bright-yellow precipitate (CdS); insoluble in Am_2S, in KHO, and in KCy solution; soluble in boiling dilute HNO_3 and in boiling dilute H_2SO_4. CdS is reprecipitated from the solution in H_2SO_4, if the liquid is diluted and H_2S is added.

317. **Blowpipe-test.**—If powdered $CdSO_4$ is mixed with Na_2CO_3 in a cavity on charcoal and the mixture is heated in the inner blowpipe-flame, a brown incrustation is obtained on the charcoal.

DETECTION AND SEPARATION OF METALS IN GROUP II.A.

318. The procedure drawn up in Table II.A (**512**) depends upon the following differences shown by the metals of this Group.

1. The solubility of Bi_2S_3, CuS, and CdS in strong boiling HNO_3, in which HgS and PbS are insoluble and remain as HgS and $PbSO_4$ respectively after treatment with the acid.

2. The solubility of $PbSO_4$ in a mixture of HĀ with excess of AmHO, in which HgS is insoluble.

3. The solubility of CuH_2O_2 and CdH_2O_2, and the insolubility of BiH_3O_3, in excess of AmHO.

4. The solubility of CdS and insolubility of CuS in boiling dilute H_2SO_4: or the insolubility of CdS in KCy solution, in which CuS easily dissolves.

319. A Solution which may contain the Metals of Group II.A is first made acid, if it is not already so, with a little HCl. Whether the liquid is clear or not, H_2S is then passed into it for several minutes. The precipitate is filtered off, and the clear filtrate is diluted with a little water and H_2S is passed through it again for a short time. This dilution and treatment with H_2S are repeated until no further precipitate is produced in the filtrate; it may then be rejected. The whole of the precipitate which has been caused by the action of H_2S is then examined by Table II.A, Copper Group (**512**).

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GROUP II.B.—ARSENIC GROUP.

320. This Group includes As, Sb, Sn.

Its members differ from those of Groups III.A, III.B, IV., and V. by being precipitated as sulphides by H_2S from a solution which has been made acid with HCl. They differ from those of Group I. by not being precipitated by HCl.

The sulphides of this Group differ from those of Group II.A by being soluble in Am_2S and in KHO.

Each metal of this Group forms two series of compounds, which resemble each other in many reactions, but also present certain differences. The two classes are distinguished conveniently by the terminations *-ic* and *-ous*; thus we speak of arsen*ic* acid and arseni*ous* acid.

ARSENIC (As).

Use for an arseni*ous* compound arsenious oxide (As_2O_3), dissolved by boiling water; and for an arsen*ic* compound sodium hydrogen arsenate ($Na_2HAsO_4.12H_2O$), dissolved in water.

321. The tests described in the following paragraphs, except 330, give the same ultimate results with both arseni*ous* and arsen*ic* compounds. As a rule, however, the reaction proceeds slowly with an arsen*ic* compound, since the first stage consists in its conversion into an arseni*ous* compound.

Arsen*ic* compounds are converted into arseni*ous* by boiling them with H_2SO_3, with strong HCl, or with some other suitable reducing agent. Arseni*ous* compounds are oxidised into arsen*ic* compounds by being heated with HNO_3, or with some other suitable oxidising agent.

322. Hydrogen sulphide (H_2S) produces in neutral solutions of arsenious compounds only a yellow coloration; but when hydrochloric acid is added to this *cold* liquid, a light-yellow flocculent precipitate (As_2S_3) forms immediately.

Filter off some of the As_2S_3, and show that it is insoluble in boiling strong HCl, but soluble in potassium hydrate (KHO) and in ammonium sulphide (Am_2S); show also that As_2S_3 is reprecipitated from these solutions by the addition of HCL in excess.

323. In cold acidified solutions of arsen*ic* compounds H_2S produces no precipitate. But if strong HCl is added and the solution is boiled, H_2S causes in the boiling solution first a precipitate of white S and then one of yellow As_2S_3.

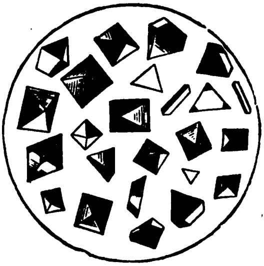
The complete precipitation of arsen*ic* solutions is rapidly effected if sulphurous acid (H_2SO_3) is first added to the solution until it smells of SO_2, and the liquid is then boiled until SO_2 is no longer smelt: the arsenious compound which is thus produced is readily and completely precipitated by H_2S from the cold solution.

324. **Reinsch's test.**—Several narrow strips of sheet copper are cleansed with sand-paper or by dipping them in strong HNO_3. They are then boiled in the arsenious solution, which has been previously acidified with dilute HCl. The surface of the copper will soon be coated with a dark-grey film of As_2Cu_5.

The strips are carefully dried by gently pressing them with filter-paper and applying a gentle heat, and are then gradually heated over a very small flame in a hot and dry test-tube. A white sublimate of As_2O_3 will form on the upper part of the tube.

A much more characteristic sublimate is obtained if moisture is entirely absent, and the sublimation is effected very slowly while the sides of the tube are hot. The sublimate is then sparkling and crystalline, and if the crystals are examined under the microscope, they will be seen to be transparent, colourless, regular octahedra and tetrahedra (fig. 65).

Fig. 65.



Crystals of As_2O_3.

The presence of As in the grey deposit upon the Cu should always be proved, since other metals may be deposited as grey films on Cu.

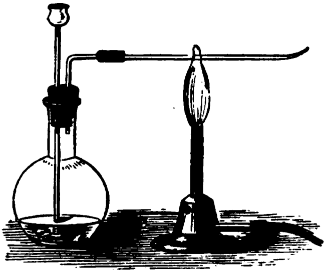
Reinsch's test serves to detect arsen*ic* compounds after their solutions have been reduced by being mixed with strong HCl or H_2SO_3 and boiled.

325. **Marsh's test.**—Fit a four-ounce flask with a thistle-funnel and a bent delivery-tube, as is shown in fig. 66.

Attach a tube of hard glass about four inches in length to the delivery-tube by means of a rubber joint, and support the tube on a ring of a retort-stand. Cover the bottom of the flask with granulated zinc, free from As and Sb; add a little water, and then pour in through the funnel-tube a little strong HCl.

After hydrogen has been evolved for some time, and has expelled all air from the flask, there will be no risk of an explosion occurring when the gas is lighted (55, Expt. 64); but it is well to cover the flask with a cloth before the hydrogen is kindled.

FIG. 66.



MARSH'S TEST FOR AS.

Now place the apparatus in a draught-closet, kindle the hydrogen, and pour some As_2O_3 solution into the flask. The flame will be seen to differ from that of pure H by its livid blue colour and by giving off white fumes of As_2O_3.

Press down upon the flame *the inside* of a porcelain-dish or crucible lid. A dusky black film of As will be deposited upon the cool surface. Pour upon this film some freshly-made solution of bleaching-powder, containing calcium hypochlorite ($CaCl_2O_2$); the arsenic stain will be rapidly dissolved.

326. **A mirror of As** will also be deposited in the glass tube if the tube is heated as is shown in fig. 66. The mirror is recognised by its appearance, by its solubility in $CaCl_2O_2$ solution, and by forming a sublimate of octahedral crystals of As_2O_3 (fig. 65), when it is slowly heated in a hot dry test-tube.

327. **Fleitmann's test.**—Boil some pieces of pure Zn in KHO solution, H will be evolved which is free from odour. If a little As_2O_3 solution is now added, and the *alkaline*

liquid is again boiled, a garlic odour will be perceptible owing to the evolution of AsH_3 :—

$$\left\{\begin{array}{l} Zn + 2KHO = H_2 + ZnK_2O_2. \\ As_2O_3 + 12H = 2AsH_3 + 3H_2O. \end{array}\right.$$

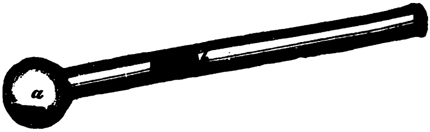
If a small piece of filter-paper, moistened with solution of silver nitrate ($AgNO_3$), is placed upon the mouth of the tube while the liquid is being boiled, the paper will be stained black by the separation of Ag.

This result is not produced by arsen*ic* compounds unless they have been first reduced by being boiled with strong HCl or with H_2SO_3.

Compounds of antimony do not give the above result; the test, therefore, distinguishes between arsenic and antimony compounds.

328. Arsenic Mirror.—Mix a little finely-powdered *dry* As_2O_3 with about four times as much freshly-ignited sodium carbonate (Na_2CO_3) and potassium cyanide (KCy). Heat this mixture in a small bulb-tube of hard glass (fig. 67), or in a small test-tube about three-sixteenths of an inch in diameter. A black mirror of arsenic will form on the cooler part of the tube (*b*), and frequently a smell of garlic may be detected at the mouth of the tube.

FIG. 67.



ARSENIC MIRROR.

If the bulb is cut off, and the glass with the mirror is gently heated in a test-tube, the As will be converted into a white sublimate of As_2O_3, or into transparent octahedral crystals of this oxide (fig. 65, page 191).

Note.—It is evident from the preceding tests that As_2O_3 is readily volatile. This is true also of As_2S_3, and of many other arsenic compounds.

329. Blowpipe-test.—Mix *a small quantity* of any solid substance containing As with Na_2CO_3, and heat the mixture on charcoal in the inner blowpipe-flame. The flame will be coloured *livid blue*, unless this tint is overpowered by the yellow sodium coloration, and a smell of garlic will be noticed when the charcoal is removed from the flame.

330. Arsenious may be distinguished from Arsenic compounds (1) by adding $AgNO_3$ solution to the perfectly neutral liquid free from chloride. *Yellow* Ag_3AsO_3 will be precipitated from arsen*ious* solutions, and *brown* Ag_3AsO_4 from arsen*ic* solutions.

(2) By adding two drops of copper sulphate ($CuSO_4$) solution, followed by excess of KOH solution; with arsen*ious* solution, a clear blue liquid will be obtained, which deposits red Cu_2O when it is boiled: no change will be produced in arsen*ic* solutions.

ANTIMONY (Sb).—Use Antimony trichloride ($SbCl_3$) solution, produced by dissolving $SbCl_3$ or Sb_2O_3 in dilute HCl.

331. **H_2S** : orange-red precipitate (Sb_2S_3). Show that the precipitate is soluble in solution of KHO or of Am_2S, and is reprecipitated from these solutions by the addition of HCl in excess: also that it is soluble in boiling strong HCl.

332. **Water (H_2O).**—If $SbCl_3$ solution is poured into much water, it yields a white precipitate or turbidity (SbOCl). This precipitate is soluble in HCl and in tartaric acid (H_2T). SbOCl differs from the similar BiOCl precipitate (306) by its solubility in H_2T.

333. **Pt and Zn.**—If the acid $SbCl_3$ solution is poured upon some platinum-foil in a porcelain-dish, and a piece of Zn is dropped upon the foil, the platinum will be rapidly coated with a black deposit of Sb. This stain is insoluble in hot HCl, but is soluble in hot HNO_3.

A slight grey or brown film or stain on the Pt is no evidence of the presence of Sb. But if the film is dissolved in hot dilute HNO_3, and this solution is diluted, then treated with H_2S and allowed to stand, a red precipitate of Sb_2S_3 will settle down from the reddish liquid, and will prove that Sb is present.

334. **SbH_3** may be prepared in a similar manner to **AsH_3** (325): it is free from smell and is not poisonous. A stain produced by Sb on porcelain, in the manner already described (325), differs from the As-stain by presenting a *dead* surface and by being insoluble in $CaCl_2O_2$ solution. Also when the stain is dissolved by warming it with yellow Am_2S, and the solution is evaporated by a gentle heat, a residue of *orange-red* Sb_2S_3 remains.

335. An Sb-mirror may be obtained in a similar manner to the As mirror (326); it differs from the As-mirror by being formed much nearer to the flame, and on both sides of the heated part of the tube.

This is due to Sb being less volatile than As. The mirror may further be distinguished by the special tests already described (334). The sublimate produced by the oxidation of this mirror (328) is insoluble in boiling water: but if the sublimate is dissolved in a little boiling HCl, the solution gives an orange-red precipitate (Sb_2S_3) when H_2S is passed into it, whereas the solution of the As_2O_3 sublimate gives yellow As_2S_3 under similar conditions.

336. If a solution containing Sb is boiled with pure Zn and KHO, it does not evolve SbH_3, and the escaping hydrogen does not stain paper moistened with $AgNO_3$ solution (327). This test distinguishes As from Sb.

337. **Blowpipe-test.**—Mix a little solid substance containing Sb with Na_2CO_3 and KCy, and heat the mixture on charcoal in the inner blowpipe-flame. A white incrustation will be formed on the charcoal, and white globules of metallic Sb will be obtained which are extremely brittle. When the melted Sb is withdrawn from the flame, each globule emits white fumes and becomes coated with white crystals of Sb_2O_3.

TIN (Sn).—Stannous and Stannic compounds.

Use for Stannous salt, $SnCl_2.2H_2O$ dissolved in dilute HCl, and for Stannic salt, $SnCl_4$ dissolved in dilute HCl.

Reagent added.	Stann*ous* salts.	Stann*ic* salts.
338. **Pass H_2S:**	*Dark-brown precipitate* (SnS): readily soluble in boiling HCl and in *yellow* Am_2S, less soluble in KHO; reprecipitated by HCl from the KHO solution as brown SnS, and from the Am_2S solution as yellow SnS_2. SnS is insoluble in *colourless* Am_2S.	*Yellow precipitate* (SnS_2), often forming only when the liquid is heated: readily soluble in boiling HCl, in Am_2S (both *yellow* and *colourless*), and in hot KHO solution, reprecipitated by HCl as yellow SnS_2 from its solution in Am_2S and in KHO.
339. **Mercuric chloride ($HgCl_2$):**	*White precipitate* (Hg_2Cl_2): turns grey on being boiled if the $SnCl_2$ is in excess (293).	No precipitate.

The following reactions give ultimately the same results with stann*ic* compounds as with stann*ous* compounds.

340. Zinc (**Zn**), when it is immersed in a solution of a tin-salt which has been acidified with HCl, becomes coated with a spongy mass of Sn. If the Zn rests on platinum-foil, the Sn is deposited in a spongy state *on the Zn and does not stain the Pt black*, differing in this way from Sb. The deposition of Sn from $SnCl_2$ is much more rapid than from $SnCl_4$.

The deposit of Sn may be rubbed and washed off the Zn, and dissolved in a little boiling strong HCl. This solution of $SnCl_2$ will yield with $HgCl_2$ solution a white precipitate of Hg_2Cl_2 (**339**).

341. Blowpipe-test. — Mix any solid compound, containing Sn, with powdered Na_2CO_3 and KCy, and heat the mixture on charcoal in the inner blowpipe-flame. A slight white incrustation will be obtained, together with white metallic particles of Sn. The globules are malleable, but differ from those of Pb by not marking paper (**303**).

DETECTION AND SEPARATION OF METALS IN GROUP II.B.

342. The procedure drawn out in Table II.B (**513**) for the detection of these metals in solution depends upon the following differences:—

1. The insolubility of As_2S_3 in hot strong HCl, in which SnS, SnS_2, and Sb_2S_3 are soluble.
2. The addition of Zn and Pt to the acid solution containing Sn and Sb, when Sn is deposited on the Zn, and Sb on the Pt.

343. From an acidified solution which may contain As, Sb, and Sn, these metals are first precipitated as sulphides by passing H_2S. After the sulphides have been filtered off, the warm filtrate is boiled and again tested by passing H_2S through it, in order to see whether the precipitation has been complete. When no further precipitation is produced by H_2S, the precipitate is examined by Table II.B, Arsenic Group (**513**).

GROUP I.—SILVER GROUP.

344. This Group includes Pb, Ag, Hg′ (mercurous compounds).

The compounds of these metals differ from those of all other common metals, by being precipitated as chlorides by HCl or by a soluble chloride. Since, however, $PbCl_2$ is somewhat soluble in water, Pb is not entirely precipitated as chloride in this Group, and is also found in Group II.A as PbS.

Lead (Pb).

345. The reactions of Pb have been already given under Group II.A in paragraphs 299–303.

Silver (Ag).—Use Silver nitrate, $AgNO_3$.

346. **Hydrochloric acid (HCl)**: white precipitate (AgCl), which becomes curdy when it is shaken or heated. This precipitate dissolves readily when it is heated with solution of ammonium hydrate (AmHO) or of potassium cyanide (KCy), and is precipitated again from these solutions when nitric acid (HNO_3) is added in excess. AgCl is insoluble in HNO_3, even on boiling; it darkens when it is exposed to sunlight or daylight in the absence of free acid.

347. **Ammonium hydrate (AmHO)**: light-brown precipitate; this precipitate is produced only when *very dilute* AmHO is added drop by drop, since it is very easily soluble in excess of AmHO.

348. **Blowpipe-test.**—If any solid substance containing Ag is mixed with powdered Na_2CO_3, and the mixture is heated on charcoal in the inner blowpipe-flame, it will give white malleable globules or scales of Ag, but no incrustation. The globules do not mark paper.

MERCUROSUM (Hg′).—Use Mercurous nitrate, $Hg'_2(NO_3)_2.H_2O$, dissolved in dilute HNO_3.

349. Reactions **293**, **294**, **297**, and **298** for mercuric salts yield precisely similar results with mercurous salts. The two following reactions (**350**, **351**), however, serve to distinguish mercuric from mercurous salts.

350. HCl: a white precipitate (Hg'_2Cl_2), which is insoluble in any single dilute acid; it is soluble in aqua regia, however, which converts it into $Hg''Cl_2$. This precipitate *becomes black when AmHO is poured upon it, but does not dissolve.*

351. AmHO, or KHO, a black precipitate, insoluble in excess.

DETECTION AND SEPARATION OF METALS IN GROUP I.

352. The Table (**510**) for the detection of the Metals in Group I. depends upon the following differences :—

1. The solubility of $PbCl_2$ in boiling water, in which AgCl and Hg'_2Cl_2 are insoluble.
2. The solubility of AgCl in AmHO, in which Hg'_2Cl_2 is insoluble.

353. A solution, which is to be examined for Pb, Ag, and Hg′, is precipitated by adding HCl in excess; it is then filtered, and the clear filtrate is tested with more HCl in order to ascertain that no further precipitate is produced. The whole of the precipitate produced by HCl is then examined by Table I. (**510**).

REACTIONS FOR ACID-RADICLES.

360. The Acid-radicles are usually detected in the course of analysis by special tests. They cannot advantageously be precipitated in Groups from which the members of each Group are then separated and identified, as is the case with the metals.

Accordingly the arrangement here adopted consists in placing together in a Group those Acid-radicles which somewhat resemble one another in their reactions; and stating at the end of each Group the differences upon which the detection of its members, when they occur together, depends.

The reactions for Acid-radicles are worked through in the same way as those for the metals, a salt or other compound containing the radicle being employed. A salt of sodium, potassium or ammonium is usually preferred for this purpose, since these metals do not as a rule interfere with the reactions of the Acid-radicles.

At the end of each Group the student may with advantage detect one or more of its members, as has been done in the case of the Metals.

GROUP I.—SULPHATE GROUP.

361. Sulphate is the only commonly occurring acid-radicle which gives, with barium chloride ($BaCl_2$), a precipitate which is insoluble in boiling hydrochloric acid (HCl).

SULPHATE—($''SO_4$).—Use Sodium sulphate, $Na_2SO_4.10H_2O$.

362. Barium chloride ($BaCl_2$), or Barium nitrate, $Ba(NO_3)_2$, gives a white precipitate of $BaSO_4$, which is insoluble when it is heated with dilute HCl or with HNO_3 (363).

363. Add $BaCl_2$ or $Ba(NO_3)_2$ to strong hydrochloric acid (HCl) or to strong nitric acid (HNO_3). A white precipitate will be produced consisting of $BaCl_2$ or $Ba(NO_3)_2$, which is insoluble in the strong acid. These precipitates are readily distinguished from $BaSO_4$ by their ready solubility in water.

364. Blowpipe-test.—When a mixture of a sulphate, or of any solid substance containing sulphur, with solid sodium carbonate (Na_2CO_3) is fused on charcoal in the inner blowpipe-flame, it yields sodium sulphide (Na_2S):—$Na_2SO_4 + 4C = Na_2S + 4CO$.

The Na_2S is readily detected by detaching the cold solid mass from the charcoal with the point of a knife, placing a portion of it upon a bright silver coin, and moistening it with a drop of water: if the moistened mass is rinsed off, after it has remained upon the coin for a short time, a black stain (Ag_2S) will be seen upon the silver where the substance rested.

Another portion of the cool mass may be moistened with a drop of HCl: a fetid smell of H_2S will be observed, and a piece of paper moistened with $Pb\bar{A}_2$ solution and held above it will be blackened.

Note.—This test is evidently only of value for detecting sulphate when it is known that sulphur is not present in any other form in the substance to be tested. It is also necessary that sulphur should not be introduced in the Na_2CO_3, or by the flame. Since coal-gas may contain sufficient sulphur to give the above reaction with pure Na_2CO_3, it is always advisable to employ the flame of a spirit-lamp rather than that of a gas-burner for this reaction.

365. Free Sulphuric acid (H_2SO_4) chars paper when it is streaked upon it; if the acid is dilute, the paper must be heated so as to concentrate the acid, black marks will then appear on the paper.

GROUP II.—CARBONATE GROUP.

366. The Group includes carbonate, sulphide, sulphite, thiosulphate, nitrite, and hypochlorite. A salt containing any of these acid-radicles gives off a characteristic gas when it is acted upon by hydrochloric acid (HCl).

CARBONATE ($''CO_3$).—Use Sodium carbonate, Na_2CO_3.

367.—**Hydrochloric acid (HCl)**, or almost any other acid, if it is poured upon a carbonate, causes colourless, almost inodorous carbon dioxide gas (CO_2) to escape with effervescence. This gas is recognised by its power of turning lime- or baryta-water milky by the production of an insoluble carbonate.

The test may be tried in the following ways:—

1. The gas may be liberated in a test-tube, and will remain in the tube if it is held erect, since CO_2 is much heavier than air. If the tube is now gradually sloped, the heavy gas may be poured into another test-tube containing a little lime-water, without allowing any liquid to flow out. If this tube is then closed with the thumb, and the lime-water is shaken up with the gas, the liquid will become milky.

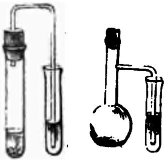
2. The test-tube or beaker in which the gas is liberated may be at once covered with a watch-glass with its convex surface downwards and with a drop of clear lime-water adhering to the middle; the drop will become milky after a time.

3. If the gas is given off in considerable quantity, it may be liberated in either of the forms of apparatus shown in fig. 68, and may be made to pass into some lime-water contained in a separate tube.

Note.—In testing for carbonate the following precautions must be attended to.

Since the milkiness which is produced in lime-water by CO_2 gradually disappears when more CO_2 is absorbed, the lime-water must be constantly watched, in order to avoid the risk of the milkiness not being observed before it has disappeared.

FIG. 68.



TESTING EVOLVED GAS.

A solid substance, which is being tested for carbonate by HCl, should be first moistened so as to drive out the air bubbles entangled in it, since the escape of these bubbles might be mistaken for a slight effervescence of CO_2.

368. **An Acid carbonate, or Bicarbonate,** is decomposed by heat into a normal carbonate, with separation of CO_2 and H_2O. Prove this by dropping some solid sodium bicarbonate ($NaHCO_3$) into boiling water in a test-tube; effervescence will occur ($2NaHCO_3 = Na_2CO_3 + H_2O$), and the gas will make lime-water milky.

369. **$MgSO_4$** gives no precipitate with a solution of bicarbonate which has been made with *cold* water; but after the solution has been boiled it will contain normal carbonate, and this will give a precipitate of white magnesium carbonate.

370. **Hydrogen carbonate, or Carbonic acid,** can only exist in dilute aqueous solution; it may be detected by boiling the liquid, and passing the CO_2, which is evolved with the steam, into lime-water; the lime-water will become milky. A bicarbonate gives the same result, but leaves a residue after evaporation.

SULPHIDE (″S).—Use solid Ferrous sulphide, FeS, and solution of Ammonium sulphide, Am_2S.

371. **HCl,** when it is poured upon a small fragment of many solid sulphides, causes hydrogen sulphide (H_2S) gas to escape with effervescence. This gas is detected by its fetid smell, resembling that of rotten eggs; and by its property of blackening solution of lead acetate ($Pb\bar{A}_2$), by the formation of PbS (see *Note*, 373).

The gas may be made to act upon the $Pb\bar{A}_2$ solution by the methods given for CO_2 (367; 2, 3); or a small strip of filter-paper, moistened with the solution, may be attached to the end of a glass rod and introduced into the gas.

372. **Silver (Ag).**—When solution of a sulphide is dropped upon a bright silver coin, it produces a black stain (Ag_2S). This stain may be removed by rubbing the coin with moist lime.

A sulphide which is insoluble in water, but gives off H_2S on the addition of HCl, produces a black stain when it is placed upon a silver coin and then moistened with a drop of HCl.

An insoluble sulphide, which does not evolve H_2S when it is heated with an acid, must be strongly heated with Na_2CO_3; the resulting mass is then extracted with water, and the solution is dropped upon the silver.

373. **$Pb\bar{A}_2$ solution** gives a black precipitate (PbS).

Note.—The solution produced by adding KHO in excess to $Pb\bar{A}_2$ solution, until the precipitate which forms at first is redissolved, is a more delicate reagent for this test, and for 371.

374. **Sulphur Sublimate.**—When sulphur is heated in a test-tube, it gives a sublimate consisting of brown drops or of yellow powder. A similar sublimate is obtained from many sulphides when they are heated in a test-tube.

375. **Sulphur dioxide evolved.** — When sulphur or a suitable sulphide is heated in a tube open at both ends and held obliquely in the flame, instead of in a test-tube, it evolves SO_2. This gas may be recognised by its pungent smell and by turning paper moistened with solution of potassium dichromate ($K_2Cr_2O_7$) green.

376. **Formation of Sulphate.**—When sulphur or a sulphide is fused with KNO_3, or is heated with strong HNO_3, it yields sulphate; the sulphate may be detected in the diluted solution by $BaCl_2$ (362, 363).

377. **Free Hydrogen sulphide (H_2S)**, either as a gas or in solution, is detected by its smell, by its action on $Pb\bar{A}_2$ solution (371), and by adding AmHO in excess and placing the liquid on bright silver (372).

SULPHITE ($''SO_3$).—Use Sodium sulphite, Na_2SO_3.

378. **HCl**, when it is poured upon a sulphite and warmed, liberates SO_2; this gas is known by its pungent smell and by turning $K_2Cr_2O_7$ solution green. A small strip of filter-paper may be wetted with the bichromate solution and then

attached by half its length to a glass rod, the other half hanging freely in the gas.

379. **Nascent Hydrogen.**—The addition of sulphite solution to a mixture of *HCl and Zn*, which is giving off hydrogen, immediately causes an evolution of H_2S. The H_2S may be detected by its smell, or by blackening a piece of filter-paper moistened with solution of $Pb\bar{A}_2$ (371).

380. **Free Sulphurous acid**, H_2SO_3, exists only in solution in water. It may be detected by its smell, and by its power of turning solution of $K_2Cr_2O_7$ bluish-green.

THIOSULPHATE, OR HYPOSULPHITE ($''S_2O_3$)—
Use $Na_2S_2O_3.5H_2O$ solution.

381. **HCl** causes no immediate change in *cold* thiosulphate solution; but the acidified liquid gradually becomes turbid by the separation of yellow S. It then contains H_2SO_3, which may be recognised by emitting the pungent smell of SO_2 when it is heated, and by adding a few drops of red $K_2Cr_2O_7$ solution, which change in colour to bluish-green.

382. **Silver nitrate** ($AgNO_3$) gives with thiosulphate solution a white precipitate ($Ag_2S_2O_3$), which gradually changes to black Ag_2S.

NITRITE ($'NO_2$).—Use KNO_2.

383. **Dilute H_2SO_4**, when it is warmed with a nitrite, causes the evolution of a reddish-brown gas with a characteristic smell. The coloured gas is best seen by placing the test-tube over a white surface.

384. **Cold $FeSO_4$ solution**, when it is added to solution of nitrite, causes the liquid to assume a dark brown colour. The colour becomes much more intense when cold dilute H_2SO_4 is added. The dark coloured substance which is produced is a compound of NO with $FeSO_4$; when the liquid is boiled, the colour disappears, and a reddish-brown gas is evolved (383).

385. **Fresh Starch solution**, if it is mixed with a few drops of KI solution, then acidified with $H\bar{A}$, and added to a nitrite, gives a deep blue colour, which is due to the liberation of iodine.

HYPOCHLORITE ('ClO).—Use Bleaching-powder solution, containing $CaCl_2O_2$.

386. **A hypochlorite** in the solid state, or in strong solution, evolves $HClO$, which resembles Cl in smell, but is less irritating to the nose.

387. **HCl, or H_2SO_4,** if it is added in excess, sets free chlorine at ordinary temperatures from a hypochlorite : this gas may be recognised by its smell and by its power of bleaching litmus :—

$$CaCl_2O_2 + 4HCl = 4Cl + CaCl_2 + 2H_2O.$$

388. **$Pb\bar{A}_2$ solution.**—If hypochlorite solution is added to $Pb\bar{A}_2$ solution, a white precipitate is formed which becomes yellow and finally brown when it is heated or allowed to stand.

DETECTION OF ACID-RADICLES IN GROUP II.

389. **The Detection of these Acid-radicles,** when they occur singly, presents no difficulty. But when they are mixed their detection is frequently a difficult problem.

The action of dilute H_2SO_4 causes the liberation of characteristic gases ; of these SO_2 may be detected by $K_2Cr_2O_7$-paper, H_2S by $Pb\bar{A}_2$-paper, Cl by litmus-paper ; and the brownish-red gas from nitrite is known by its colour and by its smell. CO_2 in the presence of much SO_2 can only be detected by passing the gases through hot $K_2Cr_2O_7$ solution which absorbs SO_2, while CO_2 passes on and may be detected by lime-water.

In some cases it is difficult to detect the acid-radicles in the presence of one another, since $HClO$, HNO_2, H_2S, and H_2SO_3 react upon each other when they are simultaneously evolved from their salts.

GROUP III.—NITRATE GROUP.

390. This Group includes nitrate and chlorate.

Salts of these acid-radicles resemble one another in many respects, more particularly in the fact that none of them are perfectly insoluble in water; hence no method of precipitating them is known.

NITRATE ('NO_3).—Use Potassium nitrate, KNO_3.

391. Ferrous sulphate and H_2SO_4.—Add to some nitrate solution, contained in a test-tube, about an equal volume of strong sulphuric acid (H_2SO_4). Mix the liquids well and cool the mixture in a stream of cold water from the tap. Then hold the tube in an inclined position, and pour some strong cold ferrous sulphate ($FeSO_4$) solution in a gentle stream down the inside. A distinct layer of the $FeSO_4$ solution should thus be formed upon the surface of the acid liquid. A *dark-brown* film or ring, which sometimes extends upwards, will appear where the two layers of liquid are in contact.

The liquids must be kept cool, since heat prevents the colour from appearing.

A mere *yellow* colour is often produced in the absence of nitrate, and may be disregarded.

392. Copper and H_2SO_4.—Place a few small scraps of Cu in some nitrate solution, and add strong H_2SO_4. Reddish-brown gas will appear at once or when the liquid is warmed. If the colour is faint it may be seen by placing the tube on a white surface.

$$8HNO_3 + 3Cu = \overline{2NO} + 3Cu(NO_3)_2 + 4H_2O.$$

NO is a colourless gas, but it yields reddish-brown NO_2 by oxidation when it is mixed with air.

The liquid becomes blue during the reaction, owing to the formation of copper nitrate in solution.

393. Reduction to Nitrite by Nascent Hydrogen.—A nitrate in solution may be reduced to a nitrite by the action of nascent hydrogen.

Add dilute H_2SO_4 to some nitrate solution, and mix it with freshly made starch-solution containing potassium iodide (KI): no colour will be produced; but when a fragment of Zn is dropped in, the liquid will assume a deep-blue colour (385).

394. Reduction to Nitrite by Ignition.—If a solid nitrate is heated to redness with fusion mixture ($Na_2CO_3 + K_2CO_3$) on platinum-foil for several minutes, a soluble alkaline nitrite will be formed, and may be extracted by boiling water. A portion of this solution may be proved to contain a nitrite by the iodine test (385). If another portion of the solution is acidified with nitric acid (HNO_3), it will be found to give no precipitate with silver nitrate ($AgNO_3$): compare this result with that obtained in the similar reaction for a chlorate (399).

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395. Blowpipe-test.—When a solid nitrate is fused on charcoal in the blowpipe-flame, the surface of the charcoal burns rapidly or *deflagrates.*

396. Ignition-test.—Certain solid nitrates evolve oxygen mixed with reddish-brown gas (NO_2) when they are heated in a glass tube closed at one end. The NO_2 is recognised by its colour and smell, and the O is detected by inflaming a glowing splinter of wood. Lead nitrate may be used in trying this experiment:—$Pb(NO_3)_2 = 2NO_2 + O + PbO$.

397. Hydrogen nitrate, or Nitric acid (HNO_3), has a strongly acid reaction. It gives with $FeSO_4$-solution a brown colour (391); and with Cu a reddish brown gas and a blue solution (392). When nitric acid is evaporated with quill-clippings, it stains them bright yellow.

CHLORATE ($'ClO_3$).—Use Potassium chlorate ($KClO_3$).

398. Strong H_2SO_4, when it is poured upon a *little* solid chlorate, becomes orange-red in colour, and evolves a bright yellow gas (ClO_2) when it is shaken:—

$$3KClO_3 + H_2SO_4 = 2ClO_2 + KClO_4 + K_2SO_4 + H_2O.$$

This gas has a smell somewhat resembling that of chlorine. It explodes readily when it is gently heated; hence, when the mixture in the tube is warmed, crackling sounds or small explosions will be produced.

Caution.—This experiment may be dangerous unless very little $KClO_3$ is used, and care should be taken that the mixture is only gently heated and that the mouth of the test-tube is turned away from the person.

399. **Reduction to Chloride by Ignition.**—Silver nitrate ($AgNO_3$) produces no precipitate with a chlorate solution which is free from chloride.

But if some solid alkaline chlorate which is free from chloride is heated in a test-tube, the salt fuses and gives off oxygen with effervescence, leaving a residue of chloride: $2KClO_3 = 2KCl + 3O_2$.

The oxygen may be detected by its power of inflaming a glowing splinter of wood (42).

The chloride in the cool residue may be dissolved by heating it with water, and the solution will give a white precipitate ($AgCl$) on addition of $AgNO_3$, which is insoluble in HNO_3 but soluble in AmHO (403).

This reaction readily distinguishes chlorate from nitrate, as will be seen by comparing it with the test for nitrate (394).

A chlorate, which does not yield a soluble chloride by ignition, must be mixed with fusion-mixture before it is heated.

400. **Blowpipe-test.**—If a chlorate is heated on charcoal in the blowpipe-flame, it causes the charcoal to deflagrate.

DETECTION OF NITRATE AND CHLORATE.

401. There is little difficulty in distinguishing nitrate from chlorate.

When salts containing these acid-radicles are present together, they more or less interfere with one another's reactions. But in the absence, or after the removal, of chloride and nitrite, nitrate and chlorate may be detected by heating the solid salts strongly for a short time with fusion mixture, and then testing for a nitrite and a chloride (394, 399).

GROUP IV.—CHLORIDE GROUP.

402. Chlorides, bromides, and iodides closely resemble one another in their reactions. They are all precipitated by $AgNO_3$ from solutions to which HNO_3 has been added in excess.

Chloride ('Cl).—Use Sodium chloride, NaCl.

403. Silver nitrate ($AgNO_3$), when it is added to the solution of a chloride, gives a *pure white* precipitate (AgCl). This precipitate collects into curdy masses when the liquid is heated or shaken: it rapidly turns violet in sunlight, and it darkens in colour slowly even in diffused daylight.

This precipitate is insoluble in HNO_3; but it is soluble in AmHO, in solution of potassium cyanide (KCy), and in solution of sodium thiosulphate ($Na_2S_2O_3$).

404. Strong Sulphuric acid (H_2SO_4), when it is warmed with a solid chloride, causes colourless, pungent hydrogen chloride gas (HCl) to be given off; this gas may be recognised by fuming in the air, by reddening moistened blue litmus-paper, and by turning a drop of $AgNO_3$ solution milky.

405. MnO_2 and H_2SO_4.—Mix some finely powdered chloride intimately with manganese dioxide (MnO_2), then add strong H_2SO_4 and warm the mixture; chlorine gas will be evolved:

$$2NaCl + MnO_2 + 3H_2SO_4 = Cl_2 + MnSO_4 + 2NaHSO_4 + 2H_2O.$$

This gas is recognised by its pale yellow colour, and by bleaching a piece of moistened litmus-paper.

Moist starch is not coloured when it is held in the escaping gas.

Note.—Since many samples of commercial MnO_2 evolve chlorine when they are heated with H_2SO_4, the MnO_2 must be carefully tested before it is used for this reaction. If it evolves Cl, the MnO_2 and H_2SO_4 must be heated together as long as any bleaching action is produced, when moist litmus is held in the tube; the substance to be tested is then added, and Cl is again tested for while the mixture is being heated.

406. **Free Hydrochloric acid** (**HCl**) evolves chlorine when it is warmed with MnO_2 (**405**), and gives a white precipitate with $AgNO_3$ (**403**).

BROMIDE ('Br).—Use Sodium bromide, NaBr.

407. **$AgNO_3$**, when it is added to solution of bromide, gives a *yellowish-white* precipitate (AgBr). This precipitate collects into curdy masses when the liquid is heated or shaken; it is insoluble in HNO_3, easily soluble in KCy solution and in $Na_2S_2O_3$ solution, and slightly soluble in AmHO. It resembles silver chloride (AgCl) in these respects, but it is distinguished by its slightly yellowish colour and by its slight solubility in AmHO.

408. **Strong Sulphuric acid** (**H_2SO_4**), when it is heated with a solid bromide, gives off pungent hydrogen bromide (HBr); the fumes are coloured *reddish-brown* by vapour of Br, which possesses a strong smell and bleaching power, and stains cold moist starch *orange-red.* If MnO_2 is mixed with the powdered bromide before the H_2SO_4 is added, Br is evolved in greater quantity and more readily (**405**).

409. **Chlorine-water**, or solution of bleaching-powder acidified with HCl, if it is added carefully drop by drop to solution of bromide, liberates Br, which colours the solution orange-red ($NaBr + Cl = Br + NaCl$). The addition of excess of Cl must be carefully avoided, since Cl combines with Br and destroys its colour.

If carbon disulphide (CS_2) is added to this solution of Br and the liquids are well shaken together, the Br will be dissolved by the CS_2: the reddish-brown solution of Br in CS_2 will sink beneath the colourless liquid, when the liquids are allowed to stand at rest.

IODIDE ('I).—Use Potassium iodide, KI.

410. **$AgNO_3$ solution,** when it is added to solution of iodide, gives a *yellow* precipitate (AgI). This precipitate is easily coagulated into curdy particles by heating or by shaking the liquid: it is insoluble in HNO_3, and very slightly soluble in AmHO, but is easily soluble in KCy solution and in $Na_2S_2O_3$ solution.

This precipitate differs from AgCl and AgBr by its distinctly yellow colour and by its slight solubility in AmHO.

411. **Strong Sulphuric acid (H_2SO_4),** when it is warmed with a solid iodide, causes pungent hydrogen iodide (HI) and violet iodine vapour to be evolved. If a rod or strip of paper moistened with freshly-made starch solution is held in the fumes it becomes blue.

Iodine is more plentifully evolved, if MnO_2 is mixed with the iodide, before the H_2SO_4 is added (405).

412. **Chlorine-water,** or solution of bleaching-powder acidified with HCl, if it is added carefully drop by drop to solution of iodide, sets iodine free. The iodine dissolves in the water, producing a brown liquid.

Note.—The addition of Cl in excess must be carefully avoided, as it would form colourless iodine chloride, which does not give the following reactions.

Divide the above brown liquid into two parts.

To one portion of the brown liquid add starch solution; an intense blue colour will be produced. When the solution is warmed it will become colourless, but it will usually regain its colour when it is cooled.

The colour will appear black unless much starch solution has been added and the liquid has been largely diluted. These conditions are also necessary in order to ensure the removal of the colour by heat.

To the other portion of the brown liquid add a drop of CS_2 and shake well; the CS_2 will dissolve the iodine, and will gradually settle down when the liquids are allowed to stand, forming a beautiful violet layer beneath the water.

413. **Nitrous acid.**—If a little potassium nitrite (KNO_2) is dissolved in solution of iodide, and a dilute acid is added, nitrous acid (HNO_2) will be produced and will liberate the iodine from the iodide. The iodine thus liberated in the solution may be identified by the methods already described (412).

Nitrous acid presents the advantage over Cl, as a means of liberating iodine from an iodide, that the addition of an excess does not hinder the detection of the iodine. The reagent is also useful because it does not liberate Br from bromide.

DETECTION OF A SINGLE ACID-RADICLE.

414. **Chloride, Bromide, or Iodide** is easily detected by the foregoing tests. The reaction with $AgNO_3$ (403) is perhaps the most characteristic for chloride, and the reaction with chlorine-water and CS_2 (409) for bromide, while iodide is detected with the greatest ease and certainty by liberating the iodine by nitrous acid in the presence of starch (413).

DETECTION OF THE ACID-RADICLES WHEN THEY ARE MIXED.

415. **The most Convenient Method** of detecting chloride, bromide and iodide, when they are present together in an aqueous solution, is described in paragraph 416.

It consists in testing for bromide and iodide in one part of the solution by the chlorine-test (409, 412); then driving off bromine and iodine, if they are present, from another part of the liquid by heating it with acidified potassium permanganate ($K_2Mn_2O_8$), and testing this liquid for chloride by means of $AgNO_3$ (403).

If a solid substance, which is insoluble in water, is to be tested, it must be boiled with Na_2CO_3 solution, and the filtered liquid must be examined, as is directed below (416).

Some substances are not decomposed by being boiled with Na_2CO_3 solution, and they require to be strongly heated with fused Na_2CO_3 in order to render the acid-radicles soluble. The cool mass remaining after the fusion is then extracted with water, and the filtered solution is subjected to the following examination.

416. **To a Portion of the Solution** under examination add a few drops of CS_2; then add cautiously, drop by drop, either chlorine-water, or acidified solution of bleaching-powder (409), shaking the liquid after each addition.

If the CS_2 does not become coloured, bromide and iodide are absent, and chloride may be tested for by adding HNO_3 in excess and $AgNO_3$ to another part of the solution (403).

If the CS_2 becomes coloured, all three acid-radicles may be present.

If the CS_2 becomes brown, **Iodide is Absent** and **Bromide is Present.**

If the CS_2 is coloured violet, **Iodide is Present.** Continue the cautious addition of the chlorine-water with constant shaking of the liquid. Either the violet colour will disappear, leaving the CS_2 colourless, when bromide is absent; or the CS_2 will become brown, showing the **Presence of Bromide.**

Test for chloride in another portion of the solution as follows. Acidify it with dilute H_2SO_4 and add a little potassium permanganate solution: a brown coloration will appear. Boil the solution in a porcelain-dish until the brown colour disappears.

Add more permanganate solution cautiously and boil the liquid, and continue these processes until the addition of the permanganate no longer produces any brown colour, and the liquid remains pale pink for a few seconds. Then add $AgNO_3$: the formation of a white precipitate will denote the **Presence of Chloride.**

GROUP V.—PHOSPHATE GROUP.

417. This Group includes phosphate and arsenate, which resemble one another in their principal reactions very closely.

PHOSPHATE ($'''PO_4$).—Use Sodium hydrogen phosphate, $Na_2HPO_4.12H_2O$.

418. **Magnesium sulphate ($MgSO_4$)** solution, to which some AmCl has been added and then a little AmHO, gives with solution of phosphate a white crystalline precipitate ($MgAmPO_4.6H_2O$). This precipitate is almost insoluble in AmHO, but is easily soluble in acids. If very little phosphate is present, the precipitate often appears only when the liquid has been well shaken and then allowed to stand for some time.

419. **Ferric chloride ($FeCl_3$)**, if it is added drop by drop, after a little $H\bar{A}$ and $Na\bar{A}$ solution, to solution of phosphate, gives a yellowish-white precipitate of $FePO_4$.

420. **Ammonium molybdate.**—When a drop of phosphate solution is added to a solution of ammonium molybdate ($AmHMoO_4$) in nitric acid, it gives a yellow precipitate. The formation of this precipitate is hastened by *gently* warming and stirring or shaking the liquid, but it often appears only after a time. The precipitate is only slightly soluble in inorganic acids, and is practically insoluble in HNO_3.

Part of the precipitate usually adheres firmly to the inside of the tube; this may be removed by AmHO solution, in which it is readily soluble.

This test is a most delicate one, if it is made with proper care.

If the solution to be tested is alkaline, it must be made distinctly acid with HNO_3. It is then to be added *in small quantity only* to some of the $AmHMoO_4$ solution in a test-tube; and a further quantity is only added if no yellow precipitate forms after a few minutes, when the liquid has been *gently* warmed and stirred.

This precaution is extremely important; since the presence of *an excess of phosphate altogether prevents the formation of the precipitate.*

421. Silver nitrate ($AgNO_3$) gives a yellow precipitate (Ag_3PO_4), which is soluble in AmHO and in HNO_3.

ARSENATE ($'''AsO_4$).—Use solution of Sodium hydrogen arsenate, $Na_2HAsO_4.12H_2O$.

422. The precipitates formed in solution of arsenate by $MgSO_4$, by $FeCl_3$, and by $AmHMoO_4$, correspond in appearance and general properties with those formed in phosphate solution by the same reagents (**418**, **419**, **420**). The precipitate, however, which $AgNO_3$ gives with an arsenate is *brown*, while that given by a phosphate is *yellow* (**421**).

DETECTION AND SEPARATION OF PHOSPHATE, ARSENATE, AND ARSENITE.

423. The Simplest Method of ascertaining which of these two Acid-radicles is present is to filter off the precipitate, which has been produced by the addition of AmCl, AmHO and $MgSO_4$, then wash it with a little cold water, and drop $AgNO_3$ upon it. If the precipitate consists of arsenate it will become *brown*: if it consists of phosphate it will become *canary yellow*.

424. The following Differences serve to Detect and Separate Phosphate and Arsenate:—

1. Arsenate yields a precipitate with $AmHMoO_4$ only when the liquid is *boiled*; whereas the corresponding precipitate with phosphate is produced by *a gentle heat.*

2. Arsenate solution which has been boiled with strong HCl gives, when H_2S is passed into the hot liquid, first a white precipitate of S and then yellow As_2S_3; phosphate solution under the same circumstances gives no precipitate.

3. In *perfectly neutral* arsenate solution $AgNO_3$ produces a *brown* precipitate; in phosphate solution it produces a *canary-yellow* precipitate.

425. If both Arsenate and Phosphate have to be tested for in a liquid, AmCl, AmHO, and $MgSO_4$ are added to it as long as any further precipitate is produced. Part of the precipitate is then filtered off and is tested at once for arsenate by washing it and dropping $AgNO_3$ solution upon it. If the precipitate turns yellow, *Phosphate alone is present.* If the precipitate turns brown, *Arsenate is present*, and must be separated from the other part of the precipitate before phosphate can be tested for. This is effected as follows.

The rest of the precipitate is filtered off and dissolved in a little boiling strong HCl; H_2S is then passed for several minutes into the hot solution, when the presence of arsenate is confirmed by the precipitation of white S followed by yellow As_2S_3. This precipitate is filtered off and H_2S is again passed into the boiling filtrate. If any further precipitate is produced, the gas must be further passed into the boiling liquid until it no longer causes a precipitate.

The liquid is then filtered, the clear filtrate is evaporated just to dryness, and the residue is dissolved in a little dilute HNO_3: a few drops of this solution are added to some $AmHMoO_4$-solution and the liquid is gently warmed: a yellow precipitate will indicate the *Presence of Phosphate* (420).

426. Arsenite may be tested for by acidifying the filtrate from the above Mg precipitate, and passing H_2S into it: yellow As_2S_3 will be precipitated at once if arsenite is present.

GROUP VI.—UNGROUPED ACID-RADICLES.

427. The following acid-radicles show no general resemblance to one another in their reactions: chromate, silicate, fluoride, borate, and permanganate.

CHROMATE ($''CrO_4$).—Use Potassium chromate, K_2CrO_4.

All chromates are more or less yellow or red in colour. A yellow normal chromate in solution becomes red when it is acidified, and is changed into a dichromate.

428. Various substances, which readily combine with oxygen, deoxidise acidified solution of chromate with production of a chromic salt: the deoxidation is accompanied by a change in colour of the solution from *orange-red* to *bright green.*

Show this by adding HCl or H_2SO_4 to some K_2CrO_4 solution, and then deoxidising the red solution of $K_2Cr_2O_7$ by one of the following methods. In every case the colour of the liquid will change to bright green.

a. Pass H_2S into the hot solution; white S will separate, owing to oxidation of the hydrogen of the H_2S by the chromate.

b. Add sulphurous acid (H_2SO_3) and warm.

c. Add alcohol and boil: the smell of aldehyde will be noticed: the aldehyde is the product of oxidation of the alcohol.

The following equation shows the reaction which occurs with H_2S:—

$$K_2Cr_2O_7 + 3H_2S + 8HCl = 2CrCl_3 + 2KCl + 7H_2O + 3S.$$

The green solution resulting from the above reactions will give the reactions for a chromic salt (**260–262**).

Since chromic acid is reduced by boiling HCl and by H_2S to chromic salt, the presence of chromate will lead to the precipitation and detection of CrH_3O_3 in the ordinary course of analysis.

429. **$AgNO_3$**: *purple-red* precipitate (Ag_2CrO_4): soluble in AmHO and in HNO_3.

430. **Lead acetate ($Pb\bar{A}_2$)**: *yellow* precipitate ($PbCrO_4$): soluble in KHO, insoluble in $H\bar{A}$.

SILICATE ($''SiO_3$).—Use Sodium silicate (Na_2SiO_3) solution, and finely ground sand (SiO_2) for a solid.

431. **HCl**, if it is added drop by drop to a solution of silicate, while stirring or shaking it, will give a gelatinous precipitate of silicic acid. If the solution is dilute, the silicic acid frequently remains in solution, hence the non-appearance of a precipitate is no proof of the absence of silicate.

432. **HCl and Evaporation to Dryness.**—If silicate is present in small quantity in dilute solution, it may be detected by acidifying the solution with HCl, then evaporating it to dryness in a porcelain-dish, and heating the residue to 100° as long as acid fumes are evolved. Insoluble SiO_2 is thus produced, and will remain undissolved as a white powder, when the dry residue is stirred and warmed with strong HCl.

During the last part of the evaporation over the flame, after the silica has separated as a gelatinous mass, spirting will occur unless the heat is lessened and the residue is stirred with a glass rod. Spirting is best avoided by finishing the evaporation upon a water-bath (p. 68) as soon as the liquid thickens.

The SiO_2 powder, which is left undissolved by HCl, is rendered visible by stirring the liquid and pouring it quickly into a glass vessel.

433. **Phosphate bead.**—When SiO_2 or a silicate is fused into a bead of microcosmic salt ($NaAmHPO_4.4H_2O$), it is

not dissolved; but the SiO_2 floats about in transparent particles in the liquid bead, and is visible as little opaque masses when the bead is cold. The particles of SiO_2 are usually most easily seen while they are in motion in the fused bead.

FLUORIDE ('F).—Use finely powdered Fluorspar, CaF_2.

The ordinary tests for fluoride depend upon the liberation of hydrofluoric acid, which is allowed to act upon a glass surface and to *etch* or corrode it.

434. **Strong Sulphuric acid**, when it is warmed with a little *finely powdered* fluoride in a test-tube, liberates hydrofluoric acid (HF). This acid etches the glass, and causes the sides of the tube to throw off the liquid when it is shaken against them, as a greasy surface throws off water.

If the cooled mixture is washed away and the inside of the tube is perfectly dried, it will be found to be dimmed. The roughness of the etched surface is easily felt by rubbing it with a glass rod.

435. **Etching a Watch-glass.**—The above test is made much more delicate, if it is carried out as follows.

The convex surface of a watch-glass is heated and is then rubbed with a piece of paraffin wax; it is thus covered with a layer of melted wax.

As soon as the wax has set, lines or characters are traced near the middle of the glass by gentle pressure with the point of a penknife. This removes the wax from these parts without scratching the glass.

The glass is then placed, with its coated surface downwards, as a cover to a platinum crucible or small leaden cup, which contains a little finely powdered fluoride and some strong sulphuric acid. After some cold water has been poured into the glass, to keep it cool and to prevent the wax from being melted, the bottom of the metal vessel is heated for a short time by a very small flame, and the covered vessel is allowed to stand for about fifteen minutes.

The watch-glass is then removed, and is cleansed by holding it above the flame and quickly wiping off the melted wax. The lines which were traced through the wax will now be seen to be etched upon the clean bright surface of the glass. Faintly-etched lines will be more easily seen when the cold surface of the glass is breathed upon.

436. **Silicon fluoride Test.**—The above methods do not serve for the detection of fluoride in the presence of silicate or of silica, since under these circumstances the action of H_2SO_4 liberates silicon fluoride (SiF_4) instead of HF.

SiF_4 possesses no power of etching glass; but it produces white fumes in moist air, and when it is passed into dilute AmHO, it yields a colourless flocculent precipitate of gelatinous silicic acid and H_2SiF_6 in solution.

BORATE (BO_3).—Use Borax, $Na_2B_4O_7.10H_2O$.

437. **Turmeric test.**—Powder some borate finely, and stir it with a little dilute HCl on a watch-glass. Immerse the lower half of a strip of turmeric paper in this liquid, and dry it on a watch-glass at a moderate heat either in the steam-oven (p. 152) or upon a water-bath (p. 68), or above a small flame. The part of the test-paper which contains borate will appear *reddish-brown*, and will become *bluish-black* when it is moistened with AmHO.

438. **Alcohol flame Coloration.**—Pour alcohol upon some finely-powdered borate in a test-tube or porcelain-dish: add a little strong sulphuric acid; then heat the mixture and kindle the spirit. The flame will show a *green edge*. This coloration is sometimes only seen when the flame is blown out after it has burnt for a time, and the spirit is then rekindled.

439. **Hydrogen borate, or Boric acid (H_3BO_3),** usually occurs combined with $2H_2O$ as a crystalline scaly hydrate. When this substance is strongly heated it yields a fusible glass consisting of B_2O_3.

Both the acid and the oxide are soluble in water and in alcohol, and when these solutions are evaporated, the acid partially escapes with the vapour of the liquid.

The solution of boric acid changes the blue colour of litmus-paper to a dull red. The acid is readily detected by the turmeric test (437) or by the alcohol flame-coloration test (438): it also gives a green flame coloration when it is held in the Bunsen flame on a platinum wire loop.

PERMANGANATE ($''Mn_2O_8$).—Use Potassium permanganate, $K_2Mn_2O_8$.

440. **Reducing Agents.**—The addition of reducing agents, such as SO_2, H_2S, and acidified KNO_2 solution, at once destroys the deep colour of the solution of a permanganate, forming a nearly colourless manganese salt.

441. KHO, when it is added in excess, causes the purple colour of the solution to change to green; the original purple colour, however, is restored again when this liquid is acidified.

442. **Heated with Acid.**—Solid permanganate, or its strong solution, gives off oxygen when it is heated with dilute H_2SO_4, and chlorine gas when it is heated with HCl: the purple colour of the permanganate at the same time disappears. If the sulphuric acid is strong, the gas is evolved with explosive violence.

SECTION XIII.

ANALYSIS OF A SUBSTANCE OF UNKNOWN COMPOSITION.

The numbers in brackets in this section refer to the paragraphs in the text in which the tests or processes to be employed by the student are fully described.

INTRODUCTORY REMARKS.

450. In detecting the Metals or Acid-radicles in a complex substance it is usual to make a few preliminary experiments. These are most satisfactorily made upon the substance in the solid state.

The substance is then dissolved in water or in acid, if it is not already in the liquid form, and the Analytical Groups to which the metals present belong are determined by adding the Group-reagents in suitable order.

In the Table on page 157 the metals are classified in their Analytical Groups. The number of the Group is placed at the head of each column, and the name of the Group, together with the name of the Group-reagent, is also given.

After the Groups to which the Metals belong have been determined, the metals are identified by making a careful examination of the Group precipitates.

The Acid-radicles which have not been already detected by the preliminary experiments, or during the examination for the Metals, are found by special tests.

Substances which are insoluble in water and in acids, or which are metallic in nature, usually require to be examined by special methods.

Traces of substances should always be looked for. This precaution especially applies to sodium, which will be detected as a minor constituent or impurity in most substances.

It should also be noted that an acid may be considered to be a hydrogen salt, and hydrogen may therefore be found instead of a metal. Further, there will be no acid-radicle present in metallic substances, in oxides, and hydroxides.

In making the analysis each result must be fully entered in the Note-book *as soon as the experiment has been performed.* The form of entry of the results of the analysis should be generally that of the Analytical Tables. An example of entry is given at the end of this Section (**539**).

PRELIMINARY EXAMINATION OF THE SUBSTANCE.

451. Before proceeding to employ the full course of analysis, it will be well to examine a few substances for Metals and for Acid-radicles by the Preliminary Tables given in paragraphs 466–487. For a detailed account of any test in these Tables the student should refer to the description which is given of it in the preceding Sections.

452. In the Preliminary Tables (**466–487**), the principal tests are marked thus—'Expt. I.' These stand in the first column.

In the second column are placed the results which may be noticed on trying these experiments; a glance through these results will forewarn the student of what has to be looked for. The main classification of the results which may be observed is indicated by black-type letters; the subsidiary divisions are marked by small numbers and italics; and the individual results are simply placed in succession in a vertical column.

Occasionally an observation is made which it is advisable to confirm. The *Confirmatory Test* is then entered in the first column just below the original observation, and is marked *Confirmatory* to distinguish it from the main experiment; its results are placed beside it in the second column.

The student must understand that any of the results which are entered in the second column may be observed.

A few salts and mixtures should be subjected to a careful Preliminary Examination by the student. The *results should be entered, as soon as they have been obtained,* in the tabular form as is shown in paragraph 539.

EXAMINATION FOR METALS IN SOLUTION.

453. After the Preliminary Examination of a substance has been completed, the substance is dissolved (489), and the Systematic Examination of its solution is commenced (495). Before examining the solution for Metals, the following general explanation should be attended to.

454. **It is necessary first to Separate the Metals into Groups** by adding the Group-reagents in such order that each reagent precipitates the metals of one Group only, leaving in solution the metals of all other Groups which may be present (203).

The first reagent added is HCl, which precipitates only the metals of Group I., and leaves the metals of Groups II., III., IV., and V. in solution. Hence if a precipitate is obtained on adding excess of HCl, it is filtered off; any member or members of Group I. will then be present upon the filter, while the members of all other Groups will remain in the filtrate.

When H_2S is passed into the filtrate,* metals of Group II. alone will be precipitated and may be filtered off.

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* Or solution, if no precipitate has been obtained, and filtration has therefore been unnecessary.

The filtrate,* after the removal of H_2S, and the conversion of all Fe into ferric salt by boiling with HNO_3, may be mixed with AmCl and excess of AmHO. The AmHO will precipitate any members of Group III.A, while members of Group III.B and Mg will remain dissolved by the AmCl.

The addition of Am_2S to the filtrate * will then separate the metals of Group III.B as sulphides.

Group IV. is precipitated from the filtrate* by the last Group-reagent, Am_2CO_3.

The filtrate* is then examined for Group V., since the members of this Group are not precipitated by any Group-reagent and may therefore still be present in the solution.

455. Addition of Reagents in Excess.—Each Group must be *completely* precipitated before the next Group-reagent is added. If any precipitate has been formed by a Group-reagent, it is therefore necessary to add that reagent *in excess* before proceeding to add the next one.

The general method for ascertaining that a Group-reagent is present in excess consists in adding a few drops more of it to the liquid, which has been rendered clear either by filtration or by letting the precipitate settle. If any further precipitate is formed, the whole of the liquid is well stirred with more of the reagent, and the clear filtrate is then again tested in the same way. This process must be repeated until no further precipitate is caused by the addition of the reagent.

In certain cases the presence of a reagent in excess is detectable by its smell, after the solution and reagent have been well mixed, and the air above the liquid has been blown out. This is the case, for example, with H_2S and with AmHO. In other cases, as for example with yellow Am_2S, the presence of an excess is indicated by the colour of the filtrate. If the reagent causes the reaction of the liquid to change from acid to alkaline or *vice versa*, test-papers may be used to detect its presence in excess.

456. Washing Group precipitates.—Before examining each Group precipitate, it is necessary that it should be washed thoroughly from adhering solution (188).

457. Evaporation before precipitating the Third Group.—If certain substances are present, such as fluorides, borates, or silicates, it is necessary to remove them by evaporation and ignition before proceeding to precipitate Group III.

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* Or solution, if no precipitate has been obtained, and filtration has therefore been unnecessary.

But if fluorides, borates, and silicates are known to be absent, it is sufficient to boil the filtrate from Group II. until it has no smell of H_2S, and then to add a few drops of strong HNO_3, and continue the boiling for several minutes, in order to completely remove H_2S and to convert ferrous into ferric salts.

The method of examining the precipitates in Group III. may be influenced by the presence of phosphate in the solution (515).

EXAMINATION FOR ACID-RADICLES IN SOLUTION.

458. The Examination for Acid-radicles (519 *et seq.*) is somewhat less systematic than that for Metals. It is usually preceded by the precipitation of the Metals which might interfere with the tests for Acid-radicles. Certain Acid-radicles cannot be detected in the Preliminary Examination, and will always be found by special tests in this fuller Examination. If no metal is present, the substance will usually be an acid or hydrogen salt.

DETECTION OF OXIDES AND HYDROXIDES.

459. **The presence of Oxides** is usually shown if a metal is found, but the substance contains no acid-radicle, and does not present either a metallic or saline appearance. Many oxides may be recognised by giving off oxygen when they are heated, but they are commonly detected by their general appearance.

460. **Hydroxides** resemble oxides in having a non-metallic appearance and in containing no acid-radicle, but they usually differ from oxides by giving off water when they are heated.

Both oxides and hydroxides which are soluble in water usually give an alkaline reaction to test-papers.

ANALYTICAL TABLES.

GENERAL COURSE OF ANALYSIS FOR SOLID AND LIQUID SUBSTANCES.

I. THE SUBSTANCE FOR ANALYSIS IS A LIQUID.

461. Test its reaction with blue litmus paper, and with turmeric paper.

Observe the colour (464), smell, and any other special property of the liquid.

462. Evaporate a few drops of the liquid upon platinum-foil, thin glass, or porcelain, smelling it occasionally:—

1. **A Residue is left.**—Evaporate a portion of the solution just to dryness in a porcelain-dish, without heating the substance after it is dry; examine the residue, if time permits, by the Preliminary Tables for Metals and Acid-radicles (466 *et seq.*). Examine the larger part of the solution for Metals by the General Table (495), paying attention to paragraph 496. Reserve the rest of the solution for the Examination for Acid-radicles by paragraphs 519 *et seq.*

2. **No Residue is left.**—The liquid must consist of some volatile substance. This may be either pure water, or water containing certain gases or volatile substances such as $(NH_4)_2CO_3$, CO_2, NH_3, HCl, H_2S, SO_2, Br, which may be easily detected by special tests.

II. THE SUBSTANCE FOR ANALYSIS IS A SOLID.

463. Carefully note the **Appearance** of the substance, using a lens, if necessary. If it is metallic and lustrous, it is usually a metal or an alloy.

Note whether it is **Crystalline or Amorphous.**

Also test its **Hardness,** and try whether it is **Magnetic.** Fe, Mn, Ni, Co, and certain of their compounds are magnetic.

Note also whether the substance possesses any characteristic **Smell or Colour.**

464. The following are some of the more commonly occurring coloured compounds:—

Blue: hydrated cupric salts, and anhydrous cobalt salts.

Green: certain ferrous salts are pale green; $CuCl_2$, salts of Ni, and certain compounds of Cr are intense green.

Yellow: HgO, normal chromates; many ferric salts are brownish-yellow.

Red: HgO, HgI_2, HgS, Pb_3O_4; dichromates are orange-red: permanganates are purple-red.

Pink: salts of Mn are delicate pink; hydrated salts of Co are reddish-pink.

Brown: Fe_2O_3, PbO_2; PbO, Bi_2O_3, CdO, are light brown.

Black: CuO, MnO_2, FeS, Co_3O_4, Fe_3O_4.

White: anhydrous ferrous and Cu salts, and many other substances.

Colourless: a large number of colourless crystalline substances are known; these, as well as many of the faintly-coloured bodies, yield *white* powders when they are crushed.

465. After the above examination has been completed, the solid substance is **reduced to the finest powder possible** by rubbing it in a mortar. If the substance is soft, an ordinary Wedgwood-mortar may be used; but hard rock-specimens and minerals require to be first crushed in a clean bright steel-mortar by blows of a hammer, and to be afterwards pulverised in an agate-mortar.

A portion of this powder is submitted to the tests described in the following **Preliminary Examinations** for **Metals and Acid-radicles,** the remainder being reserved for the fuller method of examination in solution (489 *et seq*).

Experiment.	Observation.	Inference.
466. **Expt. I.—Heat a small quantity of the substance in an ignition-tube.**	**A. The substance does not change.**	Absence of *volatile* substances, and of *water.*
	B. The substance changes.	
	1. *It changes colour :—*	
	Yellow, hot. | *White,* cold.	ZnO.
	Yellow, brown ,, | *Yellow,* ,,	PbO.
	Yellow-brown, ,, | *Yellow,* ,,	SnO_2 or Bi_2O_3.
	Black, ,, | *Brown,* ,,	Fe_2O_3.
	From Pink or Red to Black.	Mn compounds and permanganates.
	From Green or Blue to Black.	$CuCO_3$, $Cu(NO_3)_2$.
	2. *It fuses, and becomes solid again on cooling.*	Salt of alkali-metal, or possibly salt of Ba, Sr, Ca, Mg.
	3. *It sublimes.*	
467. *Confirmatory.* — The sublimate is carefully examined, if necessary with the aid of a lens.	White sublimate	$HgCl_2$, As_2O_3, NH_4.
	Black sublimate	HgS.
	Yellow sublimate	As_2S_3, HgI_2.
	Sublimate of reddish drops	Free S, or polysulphide.
	Violet vapour	Presence of I.
468. *Confirmatory.*—Heat another portion of the substance, mixed with about three times as much Na_2CO_3 in fine dry powder and a little KCy, in an ignition-tube.	Grey mirror, which, when rubbed with a splinter of wood or a glass rod, or when examined by a lens, is seen to consist of globules of Hg.	Presence of Hg.
	Blackish-brown shining mirror	,, As.
469. *Confirmatory.* — Pour a little strong KHO-solution upon some of the substance and heat to boiling.	NH_3 is given off, known by its smell and by turning moist turmeric paper brown.	,, NH_4.
	4. *The substance gives off Water.*	Presence of water, absorbed or combined.
470. *Confirmatory.*—Examine the drops of water on the sides of the tube with blue litmus and turmeric papers.	The water is alkaline	Probably NH_4 compound.
	The water is acid	Presence of a volatile acid, such as HNO_3, HCl, H_2SO_4.
	5. *Gas or Vapour is given off.* *a. The Gas is without smell.*	
471. *Confirmatory.*—Introduce a glowing splinter of wood into the tube.	It burns brightly	O, from chlorate, nitrate, peroxide, or permanganate; or N_2O.
	b. The Gas or Vapour has a smell.	
472. *Confirmatory.*—Introduce a slip of paper moistened with $K_2Cr_2O_7$ solution into the upper part of the tube.	Smell of burning sulphur, and the $K_2Cr_2O_7$ turns green.	SO_2 from combustion of free S, or from some sulphur compound.
	Reddish-brown nitrogen tetroxide, which does not colour starch-paste.	Nitrate or nitrite of a heavy metal, such as Pb, Bi, Hg.

Experiment.	Observation.	Inference.
473. **Expt. II.—Dip a clean moistened loop of platinum wire into the substance, and hold it in the Bunsen-flame.** **Then moisten with a drop of strong HCl and heat again.** **Continue the heating until no further change occurs in the colour of the flame.**	**The substance Colours the Flame:**	
	1. *Intense yellow*	Na.
	Examine the flame through the indigo-prism; a crimson colour is seen.	Presence of K or Sr, or of both.
	2. *Pale lilac*, crimson through the indigo-prism . . .	K.
	3. *Yellowish-green* . . .	Ba probably: possibly Zn.
	4. *Crimson*, same through the indigo-prism	Sr.
	5. *Orange-red*, no red seen through the indigo-prism .	Ca.
	6. *Bright green*	Boric acid, Cu.
	A blue colour is seen after moistening with strong HCl	Cu.
	7. *Blue* *Intense* . . .	$CuCl_2$, $CuBr_2$.
	Livid	As, Sb, Pb.
	8. *Pale blue*	Sn, Hg.
474. **Expt. III.—Heat the finely powdered substance, in a small cavity scooped in a piece of wood-charcoal, in the blowpipe-flame.**	A. **The substance Decrepitates, or crackles.**	NaCl, or other crystalline salt.
	B. **The substance Deflagrates, or causes charcoal to burn rapidly.**	Chlorate or nitrate.
	C. **The substance Fuses easily**, and is absorbed by the charcoal, or forms a liquid bead.	Salt of alkali-metal, or possibly salt of Ba, Sr, Ca, Mg.
	D. **An Infusible Residue** is left on the charcoal:—	
	1. *The residue is White and very Luminous whilst hot.*	Probably BaO, SrO, CaO, MgO, Al_2O_3, ZnO, or SiO_2.
475. *Confirmatory.* — Detach a portion of the cool residue, and place it upon moist turmeric paper.	The paper turns brown, showing the residue to be alkaline.	BaO, SrO, CaO, and possibly MgO.
476. *Confirmatory.*—Moisten the residue on the charcoal when cool with several drops of $Co(NO_3)_2$ solution, and heat again strongly.	A *Blue* residue, the colour of which does not disappear when the residue is intensely heated.	Al_2O_3, a phosphate, arsenate, or silicate.
	A *Pink* residue	MgO.
	A *Green* residue	ZnO.

Experiment.	Observation.		Inference.
	2. *The residue is Coloured.* Colour of bead.		
	In outer flame.	In inner flame.	
477. *Confirmatory.*—Heat a small quantity of the substance in a clear colourless borax-bead, first in the outer, then in the inner blowpipe-flame. See *Note* below.	*Green*, hot, and *blue*, cold.	*Colourless*, or *red*, cold. .	Cu.
	Blue, hot and cold.	*Blue*, hot and cold . . .	Co.
	Violet, hot, and *yellow*, cold.	*Grey* or *opaque*	Ni.
	Reddish-brown, hot, *yellow*, cold.	*Olive-green*, hot and cold . .	Fe.
	Green, hot and cold.	*Green*, hot and cold . .	Cr. See 478.
	Reddish-purple, hot and cold.	*Colourless*, hot and cold . .	Mn.
478. *Confirmatory.* — The presence of Mn and Cr may be confirmed by fusing the substance with Na_2CO_3 and KNO_3 on platinum-foil.	*Light yellow* mass on cooling . .		Cr.
	Bluish-green mass on cooling . .		Mn.
	3. *The residue is Coloured, or Metallic Scales or Globules are seen.*		
479. *Confirmatory.* — Heat also some of the substance, mixed with powdered KCy and Na_2CO_3, on charcoal in the inner blowpipe-flame.	The substance is reduced to the metallic state (184).		
	a. Without Incrustation.		
	Brilliant white metal		Ag.
	Red scales or globules		Cu.
	Grey powder attracted by a magnetised knife-blade.		Fe, Co, Ni, Mn.
	b. With Incrustation.		
	Globules.	Incrustation.	
480. *Confirmatory.* — Test the malleability of the globule, and whether it marks paper (184). See *Note* below.	*White, malleable*, not marking paper.	None, or very slight . . .	Sn.
	White, brittle.	*Orange*, hot . *Yellow*, cold .	Bi.
	White, malleable, marking paper.	*Yellow*, hot and cold . . .	Pb.
	White, brittle, giving white fumes.	*White*, close to substance .	Sb.
	None.	*Yellow*, hot . *White*, cold .	Zn.
	None.	*Reddish-brown*, easily volatilised . . .	Cd.
	None.	*White*, and white fumes; smell of garlic . .	As.

Note.—When the substance to be examined contains several bodies, they frequently more or less mask one another's reactions: hence the composition of many complex mixtures is only roughly indicated by the Preliminary Examination, and must be confirmed and established in the wet way. Many substances, more particularly minerals, however, can be completely analysed by a careful Preliminary Examination.

PRELIMINARY EXAMINATION FOR ACID-RADICLES.

481. The substance is required for this examination in the state of powder or of *strong* solution.

Note.—If several Acid-radicles are present, their reactions may be more or less perfectly concealed or altered, hence failure in obtaining a certain reaction does not in all cases necessarily prove the absence of the Acid-radicle.

Experiment.	Observation.	Inference.
482. Expt. I.—Treat some of the substance with dilute HCl, note the result, and then heat moderately.	**One or more of the following Gases may be evolved :—**	
	A colourless gas without smell, which turns lime-water milky.	**CO_2 from carbonate.**
	A gas of suffocating smell, which turns $K_2Cr_2O_7$ solution green.	**SO_2 from sulphite, or from sulphate in the presence of As_2O_3 or other reducing substance.**
	Yellow S is precipitated at the same time.	**SO_2 and S from thiosulphate.**
	A gas with fetid odour, which blackens $Pb\bar{A}_2$ solution.	**H_2S from sulphide; or possibly from sulphite or thiosulphite, if Zn or other reducing agent is present.**
	A yellowish-green gas of suffocating smell, which bleaches moist litmus paper.	**Cl from hypochlorite or from the action of HCl on an oxidising substance, such as MnO_2, chromate, or nitrate.**
	A reddish gas.	**NO_2 from nitrite.**
483. Expt. II.—Heat another portion of the substance gently with strong H_2SO_4.	**i. A gas is evolved with pungent smell, which fumes in the air, and renders $AgNO_3$ solution milky.**	**HCl, HBr, HI, HF from chloride, bromide, iodide, or fluoride. (See 484, 2, *Note.*)**
484. *Confirmatory.*—Mix some of the substance with MnO_2 *free from chloride* (*Note*, 405), add strong H_2SO_4, and warm.	**A *yellow* gas is given off with strong smell, which bleaches moist litmus paper.**	**Probably Cl from chloride.**
	***Brown* vapour is given off, which colours a little moist starch powder orange-red.**	**Br from bromide.**
	***Violet* vapour is given off, which colours a drop of starch-paste blue.**	**I from iodide.**

Experiment.	Observation.	Inference.
	2. A heavy fuming suffocating gas is evolved, which does not render $AgNO_3$-solution milky. The liquid behaves as if the glass were greasy. When the tube is washed out and thoroughly dried, the inside is seen to be corroded and dimmed.	**HF from fluoride; confirm by par. 530.** *Note.*—The HF, acting upon the silica of the glass, evolves SiF_4, which is detected by holding a moistened glass rod in the gas; SiO_2 is deposited as a gelatinous film upon it.
485. *Confirmatory.*—Drop into the hot liquid a few small pieces of copper.	3. *The acid fumes are reddish*, usually seen only on *heating* after adding a few fragments of Cu. If no brown fumes are seen, proceed to 486.	Presence of nitrate.
486. *Confirmatory.* — Boil some of the solid substance with water, or take some of the liquid substance; cool, add an equal volume of strong H_2SO_4, mix and cool, and pour cold solution of $FeSO_4$ carefully in upon the top of the acid liquid.	*A dark-brown ring* or layer forms upon the surface of the acid. This is best seen when the liquid has been cooled by immersing the test-tube in cold water.	Presence of nitrate.
	4. The acid is coloured reddish-yellow, and a yellow gas is evolved which smells like Cl and bleaches litmus: on heating the acid, explosion or crackling occurs.	Presence of chlorate. *Note.*—For the detection of nitrate and chlorate when mixed, see par. 401.
	5. The substance changes from yellow to green, O being evolved as is shown by a glowing splinter of wood.	Presence of chromate.
	6. Yellowish-green gas, with suffocating smell, which bleaches moist litmus.	Cl from chloride in the presence of MnO_2 or other oxidising substance.
	7. The same gas as in 6, but coloured *reddish-brown* by nitrous fumes: or coloured reddish-brown by $CrOCl_2$.	Chloride in presence of nitrate or nitrite. Chloride in presence of chromate.
	8. Purple fumes are given off with explosive violence.	Permanganate.

487. The S present in a sulphide, which is not decomposed by dilute HCl, is detected by fusing the powdered substance with two or three times as much fusion mixture ($Na_2CO_3+K_2CO_3$) in a covered crucible, boiling with a little water, and placing a drop of the solution upon a bright silver coin, when a dark stain will be produced (372).

EXAMINATION FOR METALS IN SOLUTION.

488. After the Preliminary Examinations have been completed, a solution of the solid substance must usually be prepared, and this solution must be submitted to the fuller and more Systematic Examination, which commences at par. 495. This is often termed "The Examination in the Wet Way."

PROCESS OF SOLUTION.

489. A portion of the solid, reduced to an impalpable powder (465), is boiled in a flask or boiling-tube with distilled water: if it dissolves entirely, proceed to par. 495.

If the powder does not dissolve, allow the liquid to stand until the undissolved portion has in great part settled, then decant through a filter: the filtrate is Solution I.:—

Residue in flask: boil with dilute HCl (492), allow any residue to settle, decant through the filter used above (493); repeat this process, and finally boil the residue with strong HCl, let settle, and decant through the filter:—			
Filtrate is	**Residue**: heat with dilute, then with strong HNO_3 (494), as was directed above for HCl; and decant through the filter:—		
	Filtrate is	**Residue** may be warmed for a short time with a mixture of strong HNO_3 (494) with three times as much HCl; if this does not dissolve it, dilute the acid mixture and decant it through the filter:—	
		Filtrate is	**Residue**: wash well with water, dry, and examine as directed for substances insoluble in water and acids (534 *et seq.*).
HCl Solution, or Solution II.	HNO_3 Solution, or Solution III.	Aqua regia Solution, or Solution IV.	

490. **The Separate Compound Substances** which are present in a **Complex Mixture** may frequently be discovered by carefully noting the action of the different solvents mentioned above.

Thus when the substance has been heated with water, the water may be tested by means of test-papers, or by evaporating a few drops upon a watch-glass in order to ascertain whether any portion of the substance has been dissolved. If anything has been dissolved this solution may be analysed separately.

Each solution may be similarly examined by evaporation, and separately analysed if necessary.

By this procedure the composition of each of the compounds which was originally present in the mixture may in many cases be ascertained.

491. Solutions I., II., III., IV. need not usually be examined separately. The following will be the usual method of procedure. Add to I. a few drops of dilute HNO_3, and if no precipitate is produced (496) acidify it with HCl; add also to solution III. some HCl. Any precipitate which is produced by HCl in these solutions is filtered off and is examined by Table I. (510). The solutions I., II., III., IV. are then mixed together (see *Note*), and any precipitate which is produced by mixing them is examined by pars. 534 *et seq.* as an insoluble substance.

Note.—It is best to boil down solutions III. and IV. respectively, in order to get rid of most of the HNO_3 which they contain (494), before mixing them with solutions I. and II.

The mixed solutions are then examined by pars. 495 *et seq.*

NOTES ON THE PROCESS OF SOLUTION.

492. Any changes which occur on the addition of HCl should be noted down. The evolution of gases will have been already observed and recorded in the Preliminary Examination (482).

493. When the hot HCl solution is cooled, crystals often separate. These usually consist of $PbCl_2$, rarely of $BaCl_2$. The crystals are filtered off and dissolved in a little boiling water. Ba is easily found by its green flame coloration. The formation of a bright yellow precipitate, on the addition of K_2CrO_4 to the solution, proves the *Presence of Pb.*

If Pb has been found, and a residue is left after the original substance has been treated with HCl, this residue may consist of $PbCl_2$. This salt may be dissolved by boiling it with water, and the use of HNO_3 in completing the solution of the substance is thus avoided (494).

494. HNO_3 seldom requires to be used in dissolving a substance. When this acid is employed, the quantity used should be as small as possible. Most of the acid should also be removed by evaporation before the solution is analysed, since in the course of analysis it decomposes H_2S with separation of S, and thus hinders the precipitation of Group II. Evaporation may sometimes be dispensed with by diluting the solution, and passing H_2S into it while it is cold.

GENERAL TABLE FOR SEPARATION OF

These lines run across both pages.

495. If the substance for analysis is a liquid with *neutral* or *alkaline* be at once examined as is directed below.

In this Table, and in the Group Tables which follow, it has been supposed tate will be produced when the reagent is added for its detection ; hence no

<table>
<tr><td colspan="3">Add to the Original Solution,* which must be acid in reaction (496), HCl in excess (455,</td></tr>
<tr><td rowspan="3">The Precipitate may contain:—
$PbCl_2$—<i>white.</i>
$AgCl$—<i>white.</i>
Hg_2Cl_2—<i>white.</i> (See 497.)
Examine the precipitate by Table I. (510).
<i>Note.</i>—When the hot HCl solution is cooled, $PbCl_2$ may separate in white crystals. The absence of Pb is not proved, if it is not precipitated in this group, since $PbCl_2$ is somewhat soluble, and Pb may therefore be found only in Group II.</td><td colspan="2">The Filtrate or Solution, which must give no further unless it is already dilute, and is then saturated with</td></tr>
<tr><td rowspan="2">The Precipitate may contain—
HgS—<i>black</i>
PbS—<i>black</i>
Bi_2S_3—<i>black</i>
CuS—<i>black</i>
CdS—<i>yellow</i>
} Insoluble in KHO and in Am_2S.
SnS—<i>brown</i>
Sn_2S_2—<i>yellow</i>
Sb_2S_3—<i>orange</i>
As_2S_3—<i>yellow</i>
} Soluble in KHO and in Am_2S.
Examine the precipitate <i>at once</i> by Table II. (511).
<i>Note.</i>—Any further precipitate which is formed when the filtrate is boiled must be added to the above precipitate (504).</td><td>The Filtrate or Solution is to that in column 2. As soon porcelain dish (see <i>Note</i>, and the liquid is evaporated a little strong HCl is poured filtered off (505).
Add a few drops of this acid Presence of Phosphate; if no
If Phosphate is present, try if in slight excess after AmCl ; not, test for Groups III.B,
If Phosphate is absent, add boil again and filter, keep-</td></tr>
<tr><td>The Precipitate may contain—
AlH_3O_3 – <i>almost colourless.</i>
CrH_3O_3—<i>light green.</i>
FeH_3O_3—<i>reddish-brown.</i>
Examine the precipitate by Table III.A (514).</td></tr>
</table>

NOTES ON THE GENERAL TABLE.

496. If the liquid under examination is alkaline or neutral in reaction, HNO_3 must be added in slight excess. If no precipitate forms, the liquid is examined by the General Table. If a precipitate is produced, more HNO_3 is added, and the liquid is heated. If a precipitate still remains, it is filtered off, and the filtrate is examined by the General Table, commencing with the addition of HCl.

The precipitate, which is caused and not dissolved by HNO_3, may consist of finely divided sulphur, which is separated as a *white* powder

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* By the 'Original Solution' is meant the solution of the substance if a solid, or the substance itself if a liquid, to which no reagent has been added.

METALS INTO GROUPS BY GROUP-REAGENTS.

reaction, it requires special treatment (496); if its reaction is *acid*, it may

that all metals are present; if a metal or group of metals is absent, no precipi-
filtration will be necessary, and the solution will be treated as a filtrate.

---

498), warm and filter:

---

precipitate with HCl, is boiled down if necessary (499), then diluted with water (501), H_2S by passing the gas through the solution (503, 504), then filtered;

---

diluted, and H_2S is again passed through it, any precipitate which forms being added as H_2S produces no further precipitate, the clear solution or filtrate is boiled in a column 2) until it ceases to smell of H_2S (504), a little strong HNO_3 is then added, to dryness † and the residue is gently ignited in the dish. When the dish is cool, upon the residue and heated, then water is added; any undissolved residue is

solution to some $AmHMoO_4$ solution and warm gently; a yellow precipitate shows the precipitate is formed with $AmHMoO_4$, **Phosphate is absent.**

a precipitate is produced in the remainder of the solution by the addition of AmHO if so, examine the turbid liquid containing the precipitate by Table III.P (515); if IV. and V. as if phosphate were absent.

to the rest of the solution AmCl, heat it to boiling, then add AmHO in excess (455), ing the liquid and precipitate as much as possible from exposure to the air (506).

<table>
<tr><td colspan="3">Add to the Filtrate or Solution, which may be coloured (507), Am_2S in excess (455), boil and filter:—</td></tr>
<tr><td rowspan="2">The Precipitate may contain—
ZnS—<i>white.</i>
MnS—<i>light pink.</i>
CoS—<i>black.</i>
NiS—<i>black.</i>
Examine this precipitate <i>at once</i> by Table III.B (516).</td><td colspan="2">Add to the Filtrate or Solution, which must be distinctly yellow or brown (508), Am_2CO_3 in excess (455), warm gently and filter:—</td></tr>
<tr><td>The Precipitate (509) may contain—
$BaCO_3$—<i>white.</i>
$SrCO_3$—<i>white.</i>
$CaCo_3$—<i>white.</i>
Examine the precipitate by Table IV. (517).</td><td>The Filtrate may contain Mg, K, Na.
Examine by Table V. (518).</td></tr>
</table>

from sulphide, or as a *yellow* powder from thiosulphate. This precipitate is recognised by its colour, and by not being separable by standing or by filtration; it may be disregarded.

Nitric acid may also precipitate gelatinous, colourless silicic acid SnO_2, or metastannic acid (497), Sb_2O_5, AgCl (all three white), SnS_2, As_2S_3 (both yellow), Sb_2S_3 (orange).

Hence, a full examination of this precipitate for all that it may contain will involve boiling the precipitate with aqua regia, then diluting and filtering. The insoluble residue is examined as a substance insoluble in water and acids (534). The aqua regia solution is added to the filtrate from the precipitate caused by HNO_3, and any precipitate caused on mixing these solutions is examined by Table I. (510).

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† If fluoride, borate, and silicate are known to be absent, the solution may be simply boiled for a few minutes instead of being evaporated to dryness.

497. Metastannic acid is precipitated from a solution of an alkaline metastannate by the addition of an acid. It forms a white precipitate which becomes *golden yellow* when it is moistened with $SnCl_2$ solution. This precipitate is further recognised by dissolving in the washing-water while it is being washed, and being reprecipitated when the washings run into the acid filtrate.

498. It must be remembered that strong HCl also precipitates a strong solution of a barium salt. This precipitate, however, dissolves, as $PbCl_2$ does, when it is heated with water, and differs in this respect from AgCl and Hg_2Cl_2.

499. If HNO_3 or aqua regia has been used in preparing the solution of the substance, or if the solution smells of Cl or SO_2, it should be boiled down considerably, until neither of the above gases is smelt when more HCl is added and the liquid is again boiled. The solution is then diluted, and H_2S is passed into it at once whether the dilution has caused a precipitate or not.

If As has been detected in the Preliminary Examination, H_2SO_3 solution should be added until the liquid smells of SO_2 after it has been boiled for a short time. The liquid is then heated for some time short of boiling, until it no longer smells of SO_2. This treatment reduces arsen*ic* and stann*ic* compounds to the arseni*ous* and stann*ous* condition.

In the case of As this reduction is to be recommended, since arsen*ic* compounds are not easily precipitated by H_2S. It is also desirable to reduce stannic compounds, since SnS_2, unlike SnS, readily runs through the filter, and being yellow in colour may be mistaken for S.

It will, of course, be necessary to ascertain by special tests (330 and 338, 339), made on the *original solution*, in which state As and Sn were originally present. If any precipitate, other than white sulphur, is produced by boiling with H_2SO_3, refer to par. 500.

500. While the liquid is being boiled with H_2SO_3, some H_2SO_4 is usually formed, and this may partially or completely precipitate Pb, Ba, Sr as white sulphates. Examine this precipitate for Ba and Sr by par. 505. Lead may be tested for by boiling the precipitate with $H\bar{A}$ and excess of AmHO, filtering, adding $H\bar{A}$ in excess and then K_2CrO_4: a yellow precipitate *shows Pb.*

501. On the addition of water, Bi, Sb, Sn may give white precipitates of their oxychlorides. These precipitates may be disregarded, since H_2S readily converts them into sulphides.

502. H_2S often causes a fine *white* precipitate of S, owing to the

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presence of certain oxidising substances, such as Cl, HNO_3, $HClO_3$, H_2CrO_4, $H_2Mn_2O_8$, H_2SO_3, $FeCl_3$. This precipitate is known by its freedom from colour, and by not being separable by subsidence or by filtration. It may be neglected. Great care must, however, be taken not to mistake SnS_2 for S; it resembles S in running through filter-paper, but is distinctly *yellow* in colour.

503. Certain changes may be observed while H_2S is being passed; they should be carefully noted. Thus the precipitates given by salts of Hg and Pb often show characteristic colours (292, 299) while H_2S is being passed. A solution coloured reddish-yellow by H_2CrO_4 becomes green, and purple H_2Mn_2O solution becomes colourless.

504. If the filtrate or solution after passing H_2S yields a yellow precipitate when it is boiled, this shows the presence of an arsen*ic* or a stann*ic* compound. In this case pass H_2S into the boiling liquid as long as it causes any further precipitate, or proceed as is directed in par. 499. Filter off this precipitate and add it to the precipitate which has been already obtained by H_2S.

505. SiO_2 and other substances may be present in the residue.

SiO_2 is distinguished by causing effervescence when it is strongly heated in a fused bead of Na_2CO_3, contained in a loop of platinum wire.

Al_2O_3, Fe_2O_3, Cr_2O_3 may have been rendered insoluble by the ignition of the residue. They may be dissolved by long-continued heating with strong HCl. This solution is added to the other solution which is to be examined for Group III. A.

$BaSO_4$ and $SrSO_4$ may also remain undissolved. They are detected by heating some of the residue strongly for a short time in the inner blowpipe-flame upon a loop of platinum wire, then moistening the loop with a drop of strong HCl, and holding it in the outer part of the Bunsen-flame. Crimson-red flashes, appearing red through the indigo-prism, prove the presence of Sr: a yellowish-green flame coloration shows Ba. A less rapid process consists in fusing the residue with alkaline carbonates (535).

506. Mn and Zn may be precipitated with FeH_3O_3, AlH_3O_3, CrH_3O_3. The precipitation of Mn is partly prevented by protecting the ammoniacal liquid against exposure to the air. But the further precaution should be taken of dissolving the precipitate, which has been produced by AmHO, in HCl; then reprecipitating it by AmHO, filtering, and adding the filtrate to the original filtrate from the Group III. A precipitate. This treatment of the precipitate should be repeated several times, if small quantities of Zn or Mn have to be looked for in the presence of Al or Fe.

507. The filtrate or solution, after the addition of AmCl and AmHO, may be coloured *blue* by Ni, *brown* by Co, or *reddish-violet* by

CrH_3O_3 dissolved in the excess of AmHO. Cr should be removed from this solution by dilution and prolonged boiling, before proceeding to precipitate Group III.B.

508. A *brown* or *dark-coloured* filtrate shows that Ni is present dissolved in the excess of Am_2S. If the filtrate is dark coloured, it should be poured into a porcelain-dish and boiled until black NiS separates, leaving a colourless liquid on standing. Water must be occasionally added, if necessary, in order to prevent evaporation to dryness.

The liquid is then passed through a small filter, and the black precipitate is proved to contain Ni by heating a portion of the black stained filter-paper in a fused colourless borax-bead. If the bead, after being heated in the outer flame, is *violet* or *brown* while hot and *yellow* when cold, and *becomes grey or opaque when it is heated in the inner flame*, Ni is certainly present and need not be further tested for in Table III.B (516).

The colourless filtrate is examined by the General Table for Groups IV. and V.

509. Part of the precipitate which has been produced by Am_2CO_3 often adheres firmly to the inside of the vessel. If this occurs, rinse the tube out several times with distilled water; then dissolve the precipitate by pouring in some hot $H\bar{A}$ and causing it to run over the inside of the tube. Add this solution to the $H\bar{A}$-solution of the Am_2CO_3-precipitate made in Table IV. (517, II.).

TABLE I.—SILVER GROUP.

510. **The Precipitate produced by HCl** may consist of $PbCl_2$, AgCl, and Hg'_2Cl_2.

The precipitate is washed on the filter five or six times with boiling water, the first portions of the washings being kept apart:—

<table>
<tr><td rowspan="2">Washings: the first portions may contain $PbCl_2$ in solution. If this is present in any quantity it will be deposited in brilliant crystals when the liquid is cooled.
Test for, or confirm the presence of Pb, by adding to the washings K_2CrO_4: a yellow precipitate shows:—
Presence of Pb.</td><td colspan="2">Residue: the residue is rinsed into a test-tube and boiled with AmHO, then filtered:—</td></tr>
<tr><td>Filtrate: add HNO_3 until the liquid is acid: a white precipitate shows:—
Presence of Ag.</td><td>Residue on the filter is black. Confirm, if necessary, as is directed for the Hg-residue in 512.
Presence of Hg'
(Mercurosum).</td></tr>
</table>

TABLE II.—COPPER AND ARSENIC GROUPS.

511. The Precipitate produced by H_2S in the acid solution is washed with hot water, and is carefully covered to protect it against being oxidised by contact with the air. It is then removed from the filter and boiled with KHO-solution, and the liquid is filtered. The undissolved residue is examined by Table II.A (512), below. The filtrate is treated as is described on page 242, and is then examined by Table II.B (513).

<table>
<tr><td colspan="5">TABLE II.A.—COPPER GROUP.</td></tr>
<tr><td colspan="5">512. The Residue may contain HgS, PbS, Bi_2S_3, CuS, CdS. Wash the precipitate until it is free from chloride, and heat it with strong HNO_3 in a porcelain dish for a few minutes. Boil off nearly all the acid, add a little dilute H_2SO_4, let the liquid stand for some time, with occasional stirring, and filter :—</td></tr>
<tr><td colspan="2" rowspan="2">Residue may contain HgS (<i>black</i>), $PbSO_4$ (<i>white</i>); but if it is <i>white</i>, Hg may still be present.
Remove the residue from the filter with a little $Am\bar{A}$-solution (190, 2), boil and filter :—</td><td colspan="3">Filtrate may contain Bi, Cu, Cd.
Add AmHO in excess, boil and filter :—</td></tr>
<tr><td rowspan="2">Precipitate Bi(OH)$_3$ is not easily seen in a dark-blue solution: wash with hot water, dissolve off the filter by pouring upon it <i>a few drops</i> of boiling dilute HCl, allow this solution to drop into much water, milkiness shows :—
Presence of Bi.
<i>Note.</i>—A precipitate produced by AmHO must always be tested as above for Bi, since traces of Pb and Hg might possibly appear here as a white precipitate and be mistaken for Bi.</td><td colspan="2">Filtrate, <i>if colourless</i>, is at once tested for Cd by passing H_2S.
<i>If blue, it contains Cu</i>: divide into two portions :—</td></tr>
<tr><td>Residue: dissolve in a little aqua regia, add AmHO in excess, then HCl in excess: warm the solution and drop into it a small bright strip of Cu; a grey deposit, becoming silver-white when it is rubbed, proves :—
Presence of Hg'' (Mercuricum).</td><td>Filtrate: Add $H\bar{A}$ in excess, then K_2CrO_4, a yellow precipitate :—
Presence of Pb.</td><td>To one portion add KCy-solution until the liquid is colourless, then pass H_2S to saturation: <i>yellow precipitate</i> :—
Presence of Cd.</td><td>To the other portion add $H\bar{A}$ in excess, then K_4FeCy_6-solution: reddish-brown precipitate :—
Presence of Cu.</td></tr>
</table>

TABLE II.—(*Continued*).

The Alkaline Filtrate from Group II.A may contain As_2S_3, Sb_2S_3, SnS, SnS_2. Add HCl until the liquid is acid in reaction, and pass H_2S unless the liquid smells strongly of this gas; the above sulphides, if present, will be reprecipitated; a mere milkiness due to precipitation of S may be disregarded. Examine the precipitate by the following Table (513).

<table>
<tr><th colspan="3">TABLE II.B.—ARSENIC GROUP.</th></tr>
<tr><td colspan="3">513. Filter and allow the precipitate to drain, then remove it from the filter with strong HCl (190, 2), boil and filter:—</td></tr>
<tr><td rowspan="2">The Residue is As_2S_3: wash, dry, and mix it with four times as much dry Na_2CO_3, and heat the mixture in a small bulb-tube (328); a black shining mirror confirms:—
Presence of As.
(See *Note* 2, below.)</td><td colspan="2">Filtrate may contain Sb and Sn. Place in a porcelain-dish a small stick of zinc free from Sn upon a piece of platinum-foil, pour in the acid filtrate, and allow the dish to stand until effervescence ceases.</td></tr>
<tr><td>A Black Stain on the Platinum-foil indicates presence of Sb. Dissolve the stain in a little dilute HNO_3, dilute and pass H_2S; an orange-red precipitate confirms the:—
Presence of Sb.</td><td>A Deposit on the Zinc probably consists of tin. Rub off the deposit and dissolve it in strong HCl, dilute and add $HgCl_2$ solution: a white or grey precipitate indicates:—
Presence of Sn.
(See *Notes* 1, 2, below.)</td></tr>
</table>

Note 1.—It is necessary to dissolve the deposit in HCl and to test the solution with $HgCl_2$, since impurities in the zinc are frequently mistaken for a deposit of tin.

Note 2.—Ascertain whether an arsen*ious* or arsen*ic* compound is present by testing the original solution by par. 330. Ascertain whether a stannous or stannic salt is present by testing the original solution by pars. 338, 339.

TABLE III.A.—IRON GROUP.

514. **The Precipitate produced by adding AmCl and AmHO** in excess and boiling, may contain AlH_3O_3, CrH_3O_3, Fe_3HO_3. The colour of the precipitate will usually show whether it contains any quantity of FeH_3O_3 (reddish-brown), or CrH_3O_3 (pale green), since AlH_3O_3 is colourless.

Dissolve the precipitate in *a little* boiling dilute HCl (190, 2); add to the solution pure KHO until a precipitate remains after the liquid has been stirred; then add KHO in considerable excess, boil for some time, and filter:—

<table>
<tr>
<td rowspan="2">Filtrate (<i>Note</i> 1) may contain AlH_3O_3 dissolved in excess of KHO. Add slight excess of HCl, and then slight excess of AmHO: a colourless gelatinous precipitate shows:—
Presence of Al. (See <i>Note</i> 2.)
<i>Note</i> 1.—If the alkaline solution is green, CrH_3O_3 is present: it must be precipitated by boiling the liquid in a porcelain-dish and must then be removed by filtration, before Al is tested for.
<i>Note</i> 2.—The analyst must ascertain that the Al is not derived from the KHO solution.</td>
<td colspan="2">Precipitate may contain CrH_3O_3, and FeH_3O_3. Dry the precipitate; detach it from the filter and heat it with a little fused Na_2CO_3 and KNO_3 upon platinum-foil. Continue the fusion, by heating the <i>under surface</i> of the foil, as long as any frothing occurs; a yellow mass indicates the presence of Cr (<i>Note</i> 3). Boil the platinum-foil with water until the substance is entirely dissolved or only a dark-brown powder (Fe_2O_3) remains undissolved; filter:—</td>
</tr>
<tr>
<td>Filtrate will be yellow if Cr is present (<i>Note</i> 3). Add $H\bar{A}$ in excess, boil, then add $Pb\bar{A}_2$ solution; a yellow precipitate shows:—
Presence of Cr (<i>Note</i> 4).</td>
<td>Residue in porcelain-dish; dissolve by heating with a little HCl, add some water, then a few drops of KCyS solution; a blood-red coloration shows:—
Presence of Fe (<i>Note</i> 5).</td>
</tr>
</table>

Note 3.—Traces of Mn are often precipitated with the Fe, and will be detected by imparting to the fused mass a bluish-green colour. The presence of Mn should be noted, since Mn may possibly be entirely precipitated in this Group, and will then not be detected in the next Group.

The green colour, due to the presence of Mn, conceals the pale-yellow colour due to Cr. But when the cool mass is dissolved in water and the solution is boiled with $H\bar{A}$, the manganese coloration is destroyed. After any dark-coloured precipitate has been removed by filtration, the yellow colour due to Cr becomes visible.

Note 4.—Cr may have been originally present as chromic salt, Cr replacing hydrogen in an acid; or as chromate, Cr being united with oxygen to form an acid-radicle.

Ascertain in which state Cr was present by boiling some of the original substance with Na_2CO_3 solution and filtering. A yellow filtrate, which, when it is acidified with $H\bar{A}$ and mixed with $Pb\bar{A}_2$ solution, gives a yellow precipitate, shows that Cr was *present as chromate.* If Cr was originally present as a chromic salt, it will remain undissolved as green CrH_3O_3, and will not colour the filtrate: it is found in the above Table.

Note 5.—Fe is always detected here as ferric salt; since, even if it was originally present as ferrous salt, it will have been converted into ferric salt when the solution was boiled with HNO_3 before the precipitation of Group III.A in the General Table.

In order to ascertain in which condition Fe was present, boil some of the original substance with HCl, filter if necessary, and divide the solution into two parts. To one portion add K_3FeCy_6, a dark-blue precipitate shows the *presence of Ferrosum*; to another portion add KCyS, a blood-red colour shows *presence of Ferricum.*

TABLE III.P.—PHOSPHATE GROUP.

515. If $'''PO_4$ has been found in the General Table in the filtrate from Group II., and a precipitate has been formed by adding to the rest of the solution AmCl and AmHO, add to the turbid liquid dilute HCl in quantity just sufficient to redissolve the precipitate. Then add a little $H\bar{A}$ and much $Am\bar{A}$, then $FeCl_3$ drop by drop with constant stirring until the liquid remains red.

Note.—If no distinct precipitate is produced by the addition of a little $FeCl_3$, add a small quantity of Am_2HPO_4, and then $FeCl_3$ until the solution is reddened.

Boil the turbid liquid in a flask or dish for a few minutes, and filter it while it is hot.

<table>
<tr>
<td colspan="2">The Precipitate may be rejected if $FeCl_3$ has produced a precipitate after $Am\bar{A}$ has failed to do so. If this is not the case, dry it on the filter; then remove it and heat it with a fused mixture of Na_2CO_3 and KNO_3 on platinum-foil. Boil the cool mass with water and filter.</td>
<td rowspan="2">The Filtrate should be free from $'''PO_4$, and should give no precipitate on the addition of AmCl and AmHO.
Examine it for Groups III.B, IV., and V. in the usual way (495 and 516, 517, 518).</td>
</tr>
<tr>
<td>The Residue is Fe_2O_3, and may be neglected.
Test the HCl-solution of the original substance for Fe'' and Fe''' with K_3FeCy_6 and KCNS (259).</td>
<td>Filtrate. If the filtrate is yellow, acidify a portion with $H\bar{A}$, and add $Pb\bar{A}_2$. A yellow precipitate shows:—
Presence of Cr.
Add to the rest of the filtrate AmCl in excess, and allow the liquid to stand: a white, gelatinous precipitate shows:—
Presence of Al.</td>
</tr>
</table>

TABLE III.B.—ZINC GROUP.

516. **The Precipitate produced by Am_2S** in the solution, or in the filtrate from Group III.A, may contain ZnS, MnS, NiS, CoS. The colour of the precipitate will usually show

whether it contains NiS or CoS which are black, or consists only of MnS (pink) or ZnS (white).

The presence of Ni will have been indicated by the dark-brown colour of the filtrate obtained after the Group precipitate has been boiled with an excess of yellow Am_2S. If the black precipitate, obtained by boiling the dark-brown filtrate (508), has also been proved to be NiS by the colour of the borax-bead, the further tests for Ni in the following Table may be omitted.

Remove the precipitate from the filter with cold dilute HCl (190, *c*), allow the liquid to stand, occasionally stirring it well with the precipitate. The precipitate will either dissolve, leaving only white sulphur, in which case the milky liquid may be at once examined by III. (p. 246); or a black residue will be left, which must be filtered off and examined by I. (below), the filtrate being examined by III. (p. 246) :—

I. The Residue is Black, and may contain NiS, CoS.

Heat a small quantity in a fused borax-bead in the extreme tip of the outer blowpipe-flame, and note the colour of the bead: then fuse the bead for some time in the inner blowpipe-flame, and again observe its colour. Any one of the following observations may be made:—

Blue bead in both flames shows:— **Presence of Co.** Co need not be further tested for; but if any doubt exists as to the presence of Ni, the rest of the residue must be examined as is directed below (II.).	**Brown or yellow bead,** in the outer flame, which becomes **grey or opaque** in the inner flame, shows **presence of Ni, absence of Co.** Unless mere traces of Co have to be tested for, no further examination need be made.	**A bead which is neither brown nor blue,** but of some intermediate tint in the outer flame, indicates the probable presence of both Co and Ni. Examine the remainder of the black residue as is directed below (II.) for Ni and Co, or for Co alone if Ni has been already detected (508).

II. Further Examination of the Residue.

Heat the residue with a little strong HCl until it is dissolved, adding occasionally a small crystal of $KClO_3$: boil down the solution nearly to dryness; a blue liquid shows **Presence of Co.** Dilute with a little water, filter if necessary, add KCy-solution slowly until the precipitate formed at first is just redissolved, boil briskly for several minutes, make decidedly alkaline with NaHO and add much Br-water; heat nearly to boiling, and allow the liquid to stand for at least ten minutes, filter:—

Precipitate (black): wash, and confirm the presence of Ni by heating some of the precipitate, or the paper stained with the precipitate, in a fused borax-bead in the outer and inner blowpipe-flames: a bead *yellow* when cold, becoming *black* in the inner flame shows:— **Presence of Ni.**	**Filtrate** must be warmed with more Br-water, and filtered from any additional precipitate which may form; then evaporated to dryness, and strong HNO_3 poured upon the residue as long as any frothing is caused; this is then evaporated just to dryness, the residue dissolved in water, excess of KHO added to it, and any precipitate filtered off and fused into a borax-bead, taking the paper stained by the precipitate if the precipitate is not easily removed: **a blue bead shows:—** **Presence of Co.**

TABLE III.B.—(*Continued*).

III. The HCl Solution or Filtrate may contain Zn, Mn.
Boil the liquid until it ceases to smell of H_2S, add a crystal of $KClO_3$, and boil for several minutes. Add pure NaHO to the cool liquid until it is alkaline, then add more NaHO, stir well and filter :—

Precipitate is white at first, but rapidly darkens in the air. Heat a portion of it with fused Na_2CO_3 and KNO_3 on platinum-foil; a bluish-green mass shows :— **Presence of Mn.** *Note.*—The Mn may be derived from permanganate.	**Filtrate**; pass H_2S, a white precipitate forms, often somewhat discoloured, showing :— **Presence of Zn.** *Note.*—If this precipitate is discoloured, it should be dissolved in a little boiling $H\bar{A}$. *White* ZnS will be precipitated by H_2S from this solution.

TABLE IV.—BARIUM GROUP.

517. The Precipitate produced by Am_2CO_3 may consist of $BaCO_3$, $SrCO_3$, and $CaCO_3$. Test the precipitate first by the flame coloration (I.) and then by reagents (II.).

I. Flame Coloration.—Dissolve a small portion of the precipitate in a few drops of strong HCl on a watch-glass, and hold a drop of this solution on a loop of platinum wire in the Bunsen-flame. A crimson coloration, visible through the indigo-prism, shows the *Presence of Sr*; a red coloration, not appearing crimson through the prism, *Presence of Ca*; a yellowish-green coloration, usually very persistent, *Presence of Ba.*

II. Dissolve the rest of the precipitate in the least possible quantity of $H\bar{A}$.

To a small portion of the cold solution add $CaSO_4$.

(a) *An immediate precipitate* shows presence of Ba. Examine the remainder of the solution for Sr, Ca by **A** (page 247).

(b) *A precipitate, which forms only when the solution is boiled,* shows *absence of Ba* and *presence of Sr.* Examine the remainder of the solution for Ca by **B** (page 247).

(c) *No precipitate forming even on boiling* shows *absence of Ba and Sr.* Examine the remainder of the solution for Ca by **C** (below).

A. **Add K_2CrO_4-solution in slight excess, and filter through a double filter-paper: the filtrate must be orange-red:—**		
Yellow precipitate confirms:— **Presence of Ba.**	**B.** **Solution or Filtrate may contain Sr and Ca. Dilute the solution with twice its volume of water: add dilute H_2SO_4 in excess, boil and filter:—**	
	Residue may contain Sr. Moisten it with HCl, and heat it on a platinum-wire loop in the Bunsen-flame: *Crimson coloration appearing crimson through the indigo-prism:—* **Presence of Sr.**	**C.** **Filtrate or Solution may contain Ca. Add AmHO in excess, and then $Am_2C_2O_4$-solution, warm and allow to stand: *White precipitate:—*** **Presence of Ca.**

TABLE V.—POTASSIUM GROUP.

518. The Filtrate, which remains after the addition to the original solution of all the Group-reagents in succession, may contain Mg, K, Na, and NH_4.

Since, however, ammonium compounds have been added as Group-reagents, NH_4 must be tested for by boiling a portion of the *original substance* with KHO-solution, and ascertaining whether any NH_3 is evolved. This test will, however, usually have been already made in the Preliminary Examination (469).

Proceed to examine for Group V. as is directed below.

Evaporate the filtrate from Group IV. to dryness in a porcelain dish, scrape out the solid residue upon a piece of platinum-foil. Heat this in the Bunsen-flame as long as any white fumes are seen to be given off, when the foil is removed for an instant from the flame. All ammonium compounds are thus entirely removed. If any residue remains (*Note* 1) it is dissolved by boiling the foil in a test-tube with a *small quantity* of water to which several drops of dilute HCl have been added. Divide this solution into two parts:—

<table>
<tr>
<td rowspan="3">EXAMINATION FOR Mg.
Before Mg is tested for in one portion of this solution, it is necessary to remove traces of Ba, Sr, or Ca which may possibly be present, and might be mistaken for Mg.
Add, therefore, several drops of H_2SO_4; then, whether a precipitate has formed or not, add a little AmCl, then AmHO in excess and several drops of $Am_2C_2O_4$; warm gently, and filter if necessary. Then add Na_2HPO_4, shake the liquid, and let it stand for a short time: a white crystalline precipitate shows:—
Presence of Mg.</td>
<td colspan="2">EXAMINATION FOR K AND Na.
In the other portion of the solution proceed to test for K and Na as is directed below.
Flame coloration.—Dip into the solution a loop of platinum wire which has been proved not to impart any colour to the flame. Hold the loop in the Bunsen-flame; one of the following results will be observed:—</td>
</tr>
<tr>
<td>**A Bright-yellow flame coloration, indicating:—**
Presence of Na (*Note* 2).
Examine this coloration through the indigo-prism; if it appears red, *presence of K* is proved: if no red colour is visible K is probably absent or present only in very minute quantity.</td>
<td>**A Pale lilac coloration,** appearing crimson through the indigo-prism, shows:—
Presence of K, and absence of Na.</td>
</tr>
<tr>
<td colspan="2">It is usual to confirm the above results by pouring the remainder of the solution upon a watch-glass, adding to it several drops of $PtCl_4$, and stirring well for some time; the formation of a yellow precipitate shows:—
Presence of K.</td>
</tr>
</table>

Note 1.—It is not safe to place much reliance upon an examination of the foil for the detection of a small quantity of residue; but the presence of a residue is usually indicated by a crackling noise, while the foil is cooling immediately after its removal from the flame. If any doubt is felt, the foil should be boiled with water and a drop of HCl, and the solution examined for Mg, K, and Na, as directed above.

Note 2.—A yellow coloration, more or less intense, will usually be obtained here, since most substances and reagents contain small quantities of Na. Hence the student must note the intensity of the coloration, and judge from it whether the quantity of Na is small or large. He must then enter accordingly, either "*Presence of Na,*" or "*Presence of Trace of Na.*"

EXAMINATION FOR ACID-RADICLES.

METHOD OF USING THE TABLE OF SOLUBILITY.

519. In conducting the Examination for Acid-radicles, much time and trouble may usually be saved, by considering which of these radicles can possibly be present. The preceding examination of the substance will usually have limited this number considerably; but it may be further reduced by the knowledge already obtained of the metals present, and of the solubility of the substance under analysis.

A reference to the Table of Solubility on the following page will show how this knowledge may be applied.

The Table is thus arranged. In horizontal lines at the head are placed the more commonly occurring metals, which yield salts or basic oxides. In the vertical column on the left is a list of that portion of the names of the compounds thus formed, which corresponds to the acid-radicle.

520. The Solubility of any Compound of one of the metals, which are placed at the top of the Table, is found by glancing down the vertical column which is headed by that metal. The letter indicating the solubility of the compound stands in a horizontal line with the acid-radicle portion of the name. Thus to find the solubility of zinc sulphate it is only necessary to glance down the vertical column with *Zn* at its head: on a horizontal line with *sulphate* stands the letter *w*, showing that zinc sulphate is soluble in water.

521. The Way in which this Table is Used, after the solubility of a substance under analysis has been ascertained, and the metals present have been detected, may be explained by an example. In a substance *which was entirely soluble in water*, the metals found were K, Ba, Ag. A glance down the column headed by these three metals shows at once

(*Continued on page* 251.)

TABLE OF SOLUBILITY.

522. A blank signifies that the solubility is unknown or unimportant.

	K.	*Na.*	*Am.*	*Mg.*	*Ba.*	*Sr.*	*Ca.*	*Fe″.*	*Fe‴.*	*Al.*	*Cr.*	*Zn.*	*Mn.*	*Ni.*	*Co.*
Oxide . . .	w.	w.	w.	a.	w.	w.	w-a.	a.	a.	a.i.	a.i.	a.	a.	a.	a.
Sulphide . .	w.	w.	w.	w-a.	w.	w.	w-a.	a^m.	a.	—	—	a.	a.	a^{mn}.	a^{mn}.
Chloride . .	w.	w.	w.	w.	w.	w.	w.	w.	w.	w.	a.i.	w.	w.	w.	w.
Iodide . . .	w.	w.	w.	w.	w.	w.	w.	w.	w.	—	—	w.	w.	—	—
Sulphate . .	w.	w.	w.	w.	i.	i.	w-a^m.-i.	w.	w.	w.	w.a.	w.	w.	w.	w.
Nitrate . .	w.	w.	w.	w.	w.	w.	w.	w.	w.	w.	w.	w.	w.	w.	w.
Phosphate .	w.	w.	w.	a.	a.	a.	a.	a.	a.	a.	a.	a.	a.	a.	a.
Carbonate .	w.	w.	w.	a.	a.	a.	a.	a.	—	—	—	a.	a.	a.	a.
Borate . . .	w.	w.	w.	a.	a.	a.	a.	a.	a.	a.	a.	a.	a.	a.	a.
Arsenite . .	w.	w.	w.	a.	a.	a.	a.	a.	a.	a.	a.	a.	a.	a.	a.
Arsenate . .	w.	w.	w.	a.	a.	a.	a.	a.	a.	—	—	—	—	a.	a.
Chromate . .	w.	w.	w.	w.	a.	a.	a.	—	w.	a.	a.	w.	i.	a.	—
Fluoride . .	w.	w.	w.	a.	a-i.	i.	a-i.	—	—	—	—	—	—	—	—
Oxalate . .	w.	w.	w.	a.	a.	a.	a.	a.	a.	a.	w-a.	a.	w-a.	a.	a.

	Hg″.	*Pb.*	*Bi.*	*Cu.*	*Cd.*	*Sb.*	*Sn″.*	*Sn⁗.*	*As‴.*	*Ag.*	*Hg′.*
Oxide . . .	a.	a^n.	a.	a.	a.	a^m.	a.	a.i.	w.a^m.	a^n.	a^n.
Sulphide . .	a^{mn}.	a^n.	a^n.	a^n.	a.	a^m.	a^m.	a^m.	a^n.	a^n.	a^n.
Chloride . .	w.	w.i.	w.b.	w.	w.	w.b.	w.b.	w.	w.	i.	a.i.
Iodide . . .	a.	w.a^m.	—	—	w.	—	w.	w.	—	i.	a^n.
Sulphate . .	w.b.	a.i,	w.b.	w.	w.	a.	w.	w.b.	—	w.a^n.	w.b.a^n.
Nitrate . .	w.b	w.	w.b.	w.	w.	—	—	w.	—	w.	w.b.
Phosphate .	a.	a^n.	—	a.	a.	—	a.	—	—	a^n.	a^n.
Carbonate .	a.	a^n.	a.	a.	a.	—	—	—	—	a^n.	a^n.
Borate . . .	—	a^n.	a.	a.	w.a.	—	a.	—	—	—	w.
Arsenite . .	a.	a^n.	a.	a.	—	a.	—	—	—	a^n.	a^n.
Arsenate . .	a.	a^n.	—	a.	—	a.	—	—	—	a^n.	a^n.
Chromate . .	w-a.	a^n·i.	a.	w.	—	a.	—	—	—	a^n.	a^n.
Fluoride . .	w.	a.	—	—	—	—	—	—	—	w.	—
Oxalate . .	a.	a.	a.	a.	a.	a.	a.	w.	—	a.	a.

The solubility of a compound is denoted by letters :—

w. Signifies soluble in water.

a. Soluble in the acids HCl, HNO_3, or aqua regia.

a^m. Soluble in muriatic, or hydrochloric acid.

a^n. Soluble in nitric acid.

a^{mn}. Soluble in a mixture of muriatic and nitric acids, or aqua regia, but not in either acid separately.

i. Insoluble in water and acids.

w.a. Letters thus placed together with a stop between, signify that in different states the substance shows the different solubilities denoted by the letters.

w-a. Letters connected by a hyphen indicate that the substance is only slightly soluble in the first solvent, and may therefore partially fall under the class denoted by the second letter.

w.b. Decomposed more or less by much water with formation of a basic salt, which is insoluble in water but soluble in acid.

which acid-radicles may be present. All acid-radicles might be present combined with K, since all its salts are soluble in water. But the presence of Ba in a substance soluble in water shows that SO_4, PO_4, CO_3, AsO_4, CrO_4, BO_3, and F cannot be present; since these acid-radicles form compounds with Ba which are insoluble in water. Ag excludes in addition S, Cl, Br, and I. Hence, of the more commonly occurring acid-radicles, NO_3 only need be tested for.

This example shows how much the examination for acid-radicles may often be simplified, when the metals present in a substance and its solubility are known.

DETECTION OF ACID-RADICLES IN THE PRECEDING EXAMINATIONS.

523. The Preliminary Examinations and the Examination for Metals frequently yield proof of the presence of certain acid-radicles; the presence of any acid-radicles thus detected will require no confirmation.

Thus on passing H_2S into the HCl-solution (495):—

A *reddish-yellow solution*, becoming green and depositing white sulphur, shows **Presence of Chromate.**

A *reddish-violet solution*, becoming nearly colourless and depositing white sulphur, denotes **Presence of Permanganate.**

A *yellow precipitate* of As_2S_3, appearing only when the liquid is boiled, shows **Presence of Arsenate.**

An insoluble residue of SiO_2, after evaporating to dryness the filtrate from the H_2S-group, shows **Presence of Silicate.**

And a yellow precipitate, obtained with $AmHMoO_4$ before precipitating Group III., shows **Presence of Phosphate.**

Of the Acid-radicles which remain to be tested for, some can be detected by the scheme in pars. 524, 525: others are most easily found by special tests made upon the original substance (526–533).

GENERAL SCHEME OF EXAMINATION FOR ACID-RADICLES.

524. If alkali-metals alone are present, the aqueous solution of the substance may be at once examined for acid-radicles by par 525. If other metals are present, they may be precipitated by the reagents which are added for the detection of acid-radicles, and may lead to error.

Precipitate these metals by boiling a portion of the finely-powdered substance with Na_3CO_3-solution, which must be further added to the clear liquid as long as it causes any precipitate. Then filter the liquid and divide the clear filtrate into three portions. Acidify these with HCl, HNO_3, and $H\bar{A}$ respectively; and examine them as is directed below, using a separate part for each test.

Note.—Some metals, such as Sn, Sb, Ag, and Cu, cannot be completely precipitated by boiling their solution with Na_2CO_3-solution. If they must be precipitated, the passage of H_2S, or the addition of Am_2S, will usually separate them as insoluble sulphides; the excess of Am_2S or H_2S must be removed by warming the solution before the tests are applied.

Since the analyst knows at this stage of the analysis what metals are present, it should not be difficult to separate them, or to allow for their presence, while the tests for acid-radicles are being tried.

525. **I. Portion acidified with HCl.**	*Present.*
On addition of $BaCl_2$ solution, a white precipitate insoluble on boiling (*Note* 1)	SO_4
On addition of AmCl and Am_2CO_3, a semi-transparent precipitate	SiO_3

II. Portion acidified with HNO_3. Refer to *Note* 2.	*Present.*
On addition of $AgNO_3$, a *pure white* precipitate, easily soluble in AmHO (*Note* 1)	Cl′*
On addition of $AgNO_3$, a *light yellow* precipitate, with difficulty soluble in AmHO	Br′
On addition of $AgNO_3$, a *yellow* precipitate, almost insoluble in AmHO	I′

* For separation of chloride, bromide and iodide, see pars 415, 416.

III. Portion acidified with HA.	*Present.*
On addition of PbA_2 solution, a *yellow* precipitate	CrO_4
On addition of $CaCl_2$ solution, a white gelatinous precipitate	F* probably
On addition of $FeCl_3$, a yellowish-white precipitate (528, 529)	PO_4, or AsO_4

* F will be readily detected in this precipitate, or better in the original substance by par. 530.

Note 1.—Unless pure Na_2CO_3 solution was used in preparing the solution for these tests, SO_4, Cl, and PO_4 if detected, may have been present only as impurities in the Na_2CO_3; portions of the original substance should accordingly be tested by pars. 526, 527, and 529.

Note 2.—If $AgNO_3$ yields a black precipitate, this proves the presence of sulphide,. or possibly of thiosulphate. In this case HNO_3 is added, and the liquid containing the precipitate is boiled. The black Ag_2S will thus be decomposed, leaving a milky liquid, in which any other precipitate will be readily seen after the fine sulphur has been coagulated by heating or shaking the liquid.

SPECIAL TESTS FOR CERTAIN ACID-RADICLES.

The following tests may be used for detecting a trace of an acid-radicle, or as confirmatory tests.

526. **Sulphate.**—A portion of the original substance is boiled with HCl, and the decanted or filtered liquid is tested with $BaCl_2$; a white precipitate shows:—**Presence of Sulphate.**

527. **Chloride.**—A portion of the original substance is warmed with HNO_3, the solution is decanted or filtered, and $AgNO_3$ is added to it; a *perfectly white* precipitate, easily soluble in warm AmHO, shows:—**Presence of Chloride.**

528. **Arsenate.**—This acid-radicle cannot be present unless As was detected during the examination for metals (468, 480). If As has been found amongst the metals, proceed to test for AsO_4 by adding HCl to a portion of the Na_2CO_3 solution (524) until it is acid, then AmCl, AmHO in excess, and $MgSO_4$. Filter, and pour a few drops of $AgNO_3$ solution upon the white precipitate on the filter; a change of colour to brown shows:—**Presence of Arsenate.**

529. **Phosphate.**—Boil some of the original substance with dilute HNO_3, and add a little of the clear solution to some $AmHMoO_4$ solution. Shake and stir the liquid well, and if no precipitate forms, warm *very gently*; a yellow precipitate shows:—**Presence of Phosphate.**

If AsO_4 has been detected (528), this test for PO_4 is not trustworthy. If any doubt is felt concerning the presence of PO_4, boil some of the substance with strong HCl, and test for PO_4, after the AsO_4 has been entirely separated by passing H_2S into the boiling HCl solution (425).

A precipitate produced by $AmHMoO_4$ may be dissolved in AmHO; AmCl and $MgSO_4$ are then added, the liquid is filtered, and $AgNO_3$ solution is dropped upon the precipitate; if it is phosphate, it will turn *yellow*; if it is arsenate, it will become *brown*.

530. **Fluoride.**—Poor a little strong H_2SO_4 upon the powdered substance, placed in a leaden cup or in a platinum crucible. Cover this with a watch-glass, which has been coated with paraffin wax, and has then had characters traced through the film with the point of a penknife. Now warm the crucible. If the characters are etched upon the glass (435), this shows:—**Presence of Fluoride.**

If SiO_2 is known to be present, the test must be made by conducting the gas, which is evolved when the substance is heated with strong H_2SO_4, into dilute AmHO; a deposit of gelatinous H_4SiO_4 proves:—**Presence of Fluoride.**

531. **Borate.**—Warm the original substance with HCl, dip turmeric paper into the liquid, and dry the paper at a gentle temperature. If the paper turns reddish-brown, and becomes bluish-black when it is moistened with AmHO, it indicates the **Presence of Borate.**

532. **Sulphide, Phosphide, and Arsenide** are frequently most easily detected after they have been converted into acids by oxidation. A portion of the finely-powdered substance is accordingly boiled with strong HNO_3, the solution is diluted with water, and tested for H_2SO_4 (526), H_3PO_4 and H_3AsO_4 (529, 423, 425).

533. **A Carbide** may usually be detected by evolving acetylene gas when it is acted upon by acid or by water: acetylene is recognised by its pungent smell, and by burning with a luminous smoky flame.

EXAMINATION OF SUBSTANCES INSOLUBLE IN WATER AND ACIDS.

534. **An Insoluble Substance** may contain any of the following substances. Those included in brackets, however, are soluble when they are heated with much water or acid.

As will be seen below, the colour frequently serves to indicate what substance is present.

1. **White**: $BaSO_4$, $SrSO_4$, ($CaSO_4$), $PbSO_4$, ($PbCl_2$), AgCl, SiO_2, ignited Al_2O_3, SnO_2, Sb_2O_5, Sb_2O_4, CaF_2.

2. **Dark coloured**: ignited Fe_2O_3 (dark brown or black), ignited Cr_2O_3 (dark green or black), $FeCr_2O_4$, PbO_2, and ignited $PbCrO_4$ (dark brown), C (black).

3. **Yellow**: S, AgBr, AgI, (PbI_2).

4. An insoluble silicate may also be present.

If sufficient of the substance is at disposal, a portion may be subjected to a Preliminary Examination (463 *et seq.*) for those substances only which may be present. In case the quantity of substance is small, however, the whole of it must be employed for the following examination.

TREATMENT WITH FUSED ALKALINE CARBONATES.

535. Mix the finely-powdered substance with five or six times its volume of fusion-mixture ($Na_2CO_3 + K_2CO_3$), and heat the mixture in a small covered porcelain crucible (fig. 41, p. 70).

Note.—If Pb and Ag have been proved to be absent by the preliminary tests, or by the substance not darkening with Am_2S, the porcelain crucible may be replaced by a platinum crucible or by a piece of platinum-foil. The use of platinum is preferable, since, after fusing in porcelain, small quantities of Al_2O_3 and SiO_2 will always be found. They are derived from the action of the Na_2CO_3 on the glaze of the porcelain.

Keep the mixture in a state of fusion for at least ten minutes. Then allow the crucible to cool, pour some water into it, and either allow it to stand, or boil the water, until the solid mass is loosened from the crucible. Treat this mass in a porcelain-dish with boiling water, crushing it by pressure with a pestle, if it does not quickly fall to pieces.

Allow the residue to settle, pour off the solution through a filter, and boil the residue with a little more water; pour off this water through the same filter, adding the filtrate to the former one.

Examine the undissolved residue by par. 536, and the filtrate by par. 537.

536. **The Residue on the Filter.**—Wash this well with boiling water, rejecting the washings. Then make a hole in the bottom of the filter, and wash the residue through into a test-tube, by pouring upon it a little boiling dilute HNO_3

(*Note* 1). The residue will dissolve entirely when it is boiled with the acid, if the fusion has been continued sufficiently long. Filter, if necessary, and examine the solution by the General Table (495), bearing in mind that only the metals enumerated in par. 534 are likely to be present (*Note* 2).

Note 1.—If Ag and Pb have been proved to be absent by the preliminary experiments, HCl may be used for dissolving the residue, and is preferable. If effervescence is produced by the acid, the presence of Ba, Sr, Ca, or Mg is proved.

Note 2.—If SiO_2 is present, it is best to evaporate the solution quite to dryness at this stage, using a water-bath towards the end if the substance spirts. The residue is then heated with dilute HCl and filtered, and the solution is examined by the General Table (495), omitting the evaporation before Group III.

537. **Aqueous Solution of the Fused Mass.**—Divide this solution into two parts, A and B.

A. Add HCl to one part of the solution until it is distinctly acid, and separate and detect SiO_2 by the treatment described above in *Note* 2. Examine the acid filtrate by the General Table (495) for Al. Other metals, such as Cr, Mn, Zn, Sn, Sb, whose oxides are soluble in alkalis, and may therefore pass into this solution, must also be tested for.

B. Test separate portions of the other part of the aqueous solution for the following acid-radicles as is described below. Unless the fusion-mixture was free from chloride, sulphate and phosphate, tests 1, 2, and 4 may be of little value.

1. **Chloride:** acidify with HNO_3 and add $AgNO_3$; white precipitate, easily soluble in AmHO.
2. **Sulphate:** acidify with HCl and add $BaCl_2$; white precipitate, insoluble on boiling.
3. **Chromate:** acidify with $H\bar{A}$ and add $Pb\bar{A}_2$; yellow precipitate. A chromate is also seen by the yellow colour of the solution. Cr thus detected may have been present acting as a metal or as a constituent of an acid-radicle. (*Note* 4, p. 243.)
4. **Phosphate:** acidify with HNO_3, add a few drops to some $AmHMoO_4$ solution, and warm gently; a yellow precipitate forms.

If As has been found by passing H_2S into the hot HCl-solution, PO_4 must be tested for after completely separating the As as As_2S_3 (425).

5. **Fluoride:** add HCl in excess, and warm gently to remove CO_2; then add AmHO in excess, then $CaCl_2$ as long as it produces any precipitate, and let stand for a time. Filter off, dry the precipitate and examine it for F by pouring strong H_2SO_4 upon it in a platinum crucible covered with a prepared watch-glass and seeing if it is etched (435).

EXAMINATION FOR K AND NA IN AN INSOLUBLE SOLUTION.

538. Since Na and K cannot be tested for in the substance after it has been treated with fused alkaline carbonates, a separate portion must be examined for these metals by the following process. The materials used must be perfectly free from K and Na.

The finely-powdered substance is mixed with its own weight of sublimed and crystallised NH_4Cl in powder, and with eight times its weight of pure $CaCO_3$. This mixture is gently heated in a platinum crucible for a few minutes, and is finally kept at a bright red heat for twenty or thirty minutes. The mass will not fuse as a whole, but the fused $CaCl_2$, which is formed during the reaction, will dissolve sufficient CaO to bring this into contact with the substance and decompose it.

The mixture is allowed to cool and is then boiled with water for some time after it has crumbled by the slaking of the CaO. The liquid is then filtered, and Am_2CO_3 solution is added to the filtrate until it causes no further precipitate of $CaCO_3$. The filtrate from this precipitate is evaporated considerably, and is freed from traces of Ca by the addition of $Am_2C_2O_4$. The clear solution will now contain K and Na as chlorides, if these metals were originally present. They may be tested for by the right hand side of Table V. (518).

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EXAMPLE SHOWING HOW TO ENTER THE RESULTS OF ANALYSIS OF A COMPLEX SUBSTANCE.

539. The substance given for analysis consisted of a powder containing pink, blue, white, and black particles. It smelt faintly of ammonia.

Preliminary Examination for Metals.

Experiment.	Observation.	Inference.
Expt. I. — Heated in a small dry test-tube.	Water given off which turned turmeric-paper brown.	Pres. of H_2O. Pres. of NH_4.
	Strong smell of NH_3-gas.	Pres. of NH_4.
	Substance blackened, no smell of burning.	Prob. pres. of Co, Cu, Mn.
	Slight white sublimate.	Pres. of As, NH_4, Hg.
	Brown nitrous fumes evolved, recognised by their smell.	Pres. of nitrate.
	Cl-gas evolved, found by smell and by bleaching litmus.	Pres. of Cl.
Confy.—Boiled a portion of the substance with KHO-solution.	NH_3-gas was evolved, recognised by its smell, and by turning moist turmeric-paper brown.	Pres. of NH_4.
Confy. — Heated strongly another portion of the dried substance with Na_2CO_3 in an ignition-tube.	No mirror formed.	Abs. of Hg and As.
Expt. II.—Heated a portion of the substance on a loop of platinum wire in the Bunsen-flame; moistened with HCl and heated again in the flame.	*Bright yellow* flame. The flame appeared *crimson* through the indigo-prism.	Pres. of Na. Pres. of K.
Heated for some time in the tip of the inner blowpipe-flame, moistened with HCl, and again held in the Bunsen-flame.	*Crimson* col^n. appearing *intense red* through the indigo-prism. *Bright green* col^n. with *blue* centre.	Prob. pres. of Sr. Pres. of Cu.

Experiment.	Observation.	Inference.
Expt. III.—Heated a portion of the substance on charcoal in the inner blowpipe flame.	The greater part of the substance fused readily, and was absorbed by the charcoal. Deflagration occurred. A red metallic residue remained.	Pres. of a salt of K, Na. Pres. of chlorate, nitrate. Pres. of Cu.
Confy. — Heated in a clear fused borax bead in the outer and inner blowpipe-flames.	In the outer flame a bead *green* while hot, *blue* when cold. In the inner flame *red and nearly opaque.*	Pres. of Cu.
Confy.—Heated on platinum foil with fused Na_2CO_3 and KNO_3.	No *bluish green* or *yellow* mass on cooling.	Abs. of Mn and Cr.
Confy.—Heated on charcoal in the inner blowpipe flame with fused Na_2CO_3.	Red metallic residue. A portion placed on a silver coin and moistened gave no black stain.	Pres. of Cu. Abs. of S.

Preliminary Examination for Acid-radicles.

Experiment.	Observation.	Inference.
Expt. I.—Added dilute HCl without heating.	A colourless gas was evolved, which was free from smell, and turned a drop of lime water milky.	Pres. of carbonate. Abs. of sulphite, hypochlorite, &c.
Heated to boiling.	Cl was evolved, recognised by its smell and by bleaching moist litmus-paper.	Pres. of nitrate, chlorate, or some other oxidising substance.
Expt. II—Added strong H_2SO_4.	A bright yellow chlorous gas evolved, which crackled when warmed.	Pres. of chlorate.
Confy. — Dropped in copper turnings and heated.	Reddish brown fumes evolved.	Pres. of nitrate.
Heated strongly, cooled, and rinsed out.	The tube when dry was seen not to be etched.	Abs. of fluoride.
Confy.—Boiled a portion of the substance with water, added strong H_2SO_4, cooled and poured in $FeSO_4$ solution carefully.	A brown ring formed on the surface of the acid.	Pres. of nitrate.

These lines run across both pages.

EXAMINATION FOR METALS

Boiled a portion of the substance with water; as it did not com-
upon the undissolved residue: effervescence occurred, and on boiling,
of Cl was perceived; the substance was completely dissolved. Cooled
previously to give no pp. on addition of a little dilute HNO_3:—

<table>
<tr><td rowspan="3"><i>No pp.</i>
<i>Abs. of Group I.</i>
Hg', Ag, and prob. Pb.</td><td colspan="2">Diluted with water and passed H_2S until the liquid smelt strongly</td></tr>
<tr><td rowspan="2"><i>A black pp.</i>
Exam^{d}. by Table II.</td><td><i>Filtrate</i> which gave no further pp. with
Boiled until it no longer smelt of H_2S, brown on addition of HNO_3 (<i>prob.</i>
Added a small portion of the HCl sol^{n}. then excess of AmHO, and boiled:</td></tr>
<tr><td><i>A brown pp.</i>
Exam$^{d.}$ by Table III.A.</td></tr>
</table>

EXAM$^{N.}$ OF PP. in GROUP II.—Removed from the filter into a porcelain-dish and boiled with KHO, filtered:—

<table>
<tr><td rowspan="4"><i>Filtrate</i>:— Acidified with HCl, a <i>white</i> milky liquid only: <i>Abs. of Group</i> II.B.</td><td colspan="4"><i>Pp.</i> removed from <i>filter</i> by a glass rod into a porcelain-dish and boiled with strong HNO_3 as long as any red fumes came off, added dilute H_2SO_4 and stirred well:—</td></tr>
<tr><td rowspan="3"><i>No pp.</i>:— <i>Abs. of Hg and Pb.</i></td><td colspan="3">Added to sol^{n}. excess of AmHO: blue solution (<i>pres. of Cu</i>):—</td></tr>
<tr><td rowspan="2"><i>No pp.</i>:— <i>Abs. of Bi.</i></td><td colspan="2">Divided the blue liquid into two portions:—</td></tr>
<tr><td>To one portion added KCy solution until colourless, then passed H_2S. No precipitate:— <i>Abs. of Cd.</i></td><td>Acidified the other portion with $H\bar{A}$, and added K_4FeCy_6 solution. Reddish-brown precipitate:— Pres. of Cu (Confirmed).</td></tr>
</table>

EXAM$^{N.}$ OF PP. IN GROUP III.A.

Dissolved in a little boiling dilute HCl, added pure NaHO in excess, boiled and filtered:—

<table>
<tr><td rowspan="2"><i>Filtrate</i>: added HCl in excess and then AmOH in slight excess; no pp.:— <i>Abs. of Al.</i></td><td colspan="2"><i>Pp.</i>: dried and fused on platinum-foil with Na_2CO_3 and KNO_3, boiled the colourless (<i>abs. of Cr</i>) mass when cold with water; decanted from the undissolved residue:—</td></tr>
<tr><td><i>Residue</i> in the dish dissolved by boiling with a little HCl, added several drops of KCyS: a blood-red coloration:— Pres. of Fe.</td><td><i>Sol^{n}.</i> being colourless proved abs. of Cr.
Acidified with $H\bar{A}$, boiled, and added PbA_2, no yellow pp.:— <i>Abs. of Cr.</i></td></tr>
</table>

IN THE WET WAY.

pletely dissolve, the sol^n. was decanted, and a little dilute HCl poured
Cl was smelt. Added a little strong HCl, boiled as long as any smell
this sol^n. and mixed it with the water sol^n. which had been proved

<table>
<tr><td colspan="3">of the gas, filtered :—</td></tr>
<tr><td colspan="3">H_2S was pink (<i>prob. pres. of Co</i>).
added some strong HNO_3 and boiled to dryness (the colour of the sol^n. changed to light
<i>pres. of Fe</i>), warmed the residue with HCl, it dissolved completely (<i>abs. of H_2SiO_3</i>).
to some $AmHMoO_4$ warmed; no pp.: <i>absence of $(PO_4)'''$</i>. To the rem^r. added AmCl,
filtered :—</td></tr>
<tr><td colspan="3"><i>Filtrate</i> was again pink (<i>presence of Co</i>): added much Am_2S and boiled, filtered :—</td></tr>
<tr><td rowspan="2"><i>A black pp.</i>
Exam^d. by Table III.B.</td><td colspan="2"><i>Filtrate</i> was yellow (<i>absence of Ni</i>): added Am_2CO_3, filtered :—</td></tr>
<tr><td><i>A white pp.</i>
Exam^d. by Table IV.</td><td><i>Filtrate.</i>
Exam^d. by Table V.</td></tr>
</table>

EXAM^N. OF PP. IN GROUP III.B.

Rinsed the pp. off the filter with some cold dilute HCl, stirred well, filtered :—

<table>
<tr><td rowspan="2"><i>Pp.</i> was black and had already been proved to contain no Ni; fused a portion into a clear borax-bead; bead blue in both flames :—
Pres. of Co.</td><td colspan="2"><i>Filtrate:</i> boiled until it no longer smelt of H_2S, added a crystal of $KClO_3$, boiled until the smell of Cl ceased, cooled and added pure NaHO in excess :—</td></tr>
<tr><td>No pp.
<i>Abs. of Mn.</i></td><td>Passed H_2S into the sol^n., no pp. :—
<i>Abs. of Zn.</i></td></tr>
</table>

EXAM^N. OF PP. IN GROUP IV.

1. *Exam^n. by Flame col^n.*—Dissolved a small quantity of the pp. in a few drops of HCl upon a watch-glass, dipped a loop of Pt-wire into the sol^n. and held it in the Bunsen-flame: a crimson-red colour was imparted to the flame, which appeared intense red through the indigo-prism: **Pres. of Sr.** This coloration was followed by a very persistent yellowish-green col^n.: **Pres. of Ba.**

2. *Exam^n. in the Wet way.*—Dissolved the rest of the pp. in as little boiling $\overline{HA}$ as possible; to a small part of the solution, perfectly cold, added $CaSO_4$ sol^n., an immediate pp. formed: **Pres. of Ba.**

To the remainder of the HĀ-sol^n, proved to be acid to litmus, added K_2CrO_4 until the liquid appeared red, warmed and poured through a double filter :—

<table>
<tr><td rowspan="2">Pp., which contained all the Ba present, was rejected.</td><td colspan="2">Diluted filtrate with twice its volume of water, added dilute H_2SO_4, boiled and filtered :—</td></tr>
<tr><td>White precipitate :—
Presence of Sr.
Confirmed by crimson flame coloration given by the precipitate.</td><td>To the filtrate added AmHO in excess and then $Am_2C_2O_4$; no precipitate :—
Absence of Ca.</td></tr>
</table>

EXAM$^{N.}$ OF FILTRATE FROM GENERAL TABLE FOR GROUP V.

Evaporated the filtrate to dryness in a porcelain dish, scraped out the residue upon platinum foil, and ignited strongly until white fumes ceased to appear : dissolved the residue off the foil by boiling with water to which several drops of HCl had been added, divided the sol$^{n.}$ into two unequal parts :—

To the larger portion added several drops of H_2SO_4 and boiled, then AmHO in excess and several drops of $Am_2C_2O_4$; on heating, a slight pp. formed ; filtered, and added to the clear filtrate Na_2HPO_4, a white crystalline pp. :— *Pres. of Mg.*	*Into the smaller portion* dipped a loop of platinum wire, and held it in the Bunsen flame, an intense yellow col$^{n.}$:— *Pres. of Na,* appearing crimson through the indigo-prism :— *Pres. of K.* Confirmed the pres. of K by stirring a fresh portion of the sol$^{n.}$ with $PtCl_4$ on a watch glass, a yellow pp. formed.

Metals found :—Cu, Fe, Co, Ba, Sr, Mg, Na, K, NH_4.

EXAMⁿ· FOR ACID-RADICLES.

The following acid-radicles have already been detected: CO_3, NO_3, ClO_3.

By reference to the Table of Solubilities (522), under the columns corresponding to the above metals, the only salt insoluble in acids is found to be $BaSO_4$; since Ba is present, and the substance is entirely soluble in acids, SO_4 *is absent.*

The following acid-radicles have also been proved to be absent in the general examination for metals:—

CrO_4, by the HCl-solution not becoming green on passing H_2S.

AsO_3 and AsO_4, by no pp. forming in Group II.B.

SiO_3, by no residue insol. in HCl remaining on evaporating to dryness with HCl for Group III.A.

PO_4, by testing with $AmHMoO_4$ in the HCl-sol^n· for Group III.A.

The only commonly occurring acid-radicles remaining to be specially tested for are therefore Cl, Br, I; and of these Br and I are probably absent, since no violet fumes of I or brown fumes of Br were evolved with strong H_2SO_4.

Boiled a portion of the substance with pure Na_2CO_3-sol^n· and filtered; acidified with HNO_3, and added $AgNO_3$, a perfectly white pp. easily sol. in AmOH:—**Pres. of Cl.**

Acid-radicles found:—CO_3, NO_3, ClO_3, Cl.

Found { *Metallic-radicles:* Cu, Fe, Co, Ba, Sr, Mg, Na, K, NH_4.
Acid-radicles: CO_3, NO_3, ClO_3, Cl.

APPENDIX.

LIST OF CHEMICAL ELEMENTS, WITH THEIR SYMBOLS AND ATOMIC WEIGHTS.

540. The words in brackets are the Latin names of the elements from which the symbols have been derived. (See *Note*, page 265.)

Name.	Symbol.	Atomic Weight.	Name.	Symbol.	Atomic Weight.
Aluminium	Al	27·1	Neodymium	Nd	143·6
Antimony (stibium)	Sb	120	Nickel	Ni	58·7
Argon	A	40	Niobium	Nb	94
Arsenic	As	75	Nitrogen	N	14
Barium	Ba	137·4	Osmium	Os	191
Beryllium	Be	9·1	Oxygen	O	16
Bismuth	Bi	208·5	Palladium	Pd	106
Boron	B	11	Phosphorus	P	31
Bromine	Br	80	Platinum	Pt	194·8
Cadmium	Cd	112·4	Potassium (kalium)	K	39·15
Cæsium	Cs	133	Praseodymium	Pr	140·5
Calcium	Ca	40	Rhodium	Ro	103
Carbon	C	12	Rubidium	Rb	85·4
Cerium	Ce	140	Ruthenium	Ru	101·7
Chlorine	Cl	35·45	Samarium	Sa	150
Chromium	Cr	52·1	Scandium	Sc	44·1
Cobalt	Co	59	Selenion	Se	79·1
Copper (cuprum)	Cu	63·6	Silicon	Si	28·4
Erbium	Er	166	Silver (argentum)	Ag	107·9
Fluorine	F	19	Sodium (natrium)	Na	23
Gallium	Ga	70	Strontium	Sr	87·6
Germanium	Ge	72	Sulphur	S	32
Gold (aurum)	Au	197·2	Tantalum	Ta	183
Helium	He	4	Tellurion	Te	127
Hydrogen	H	1	Terbium	Tr	160
Indium	In	114	Thallium	Tl	204·1
Iodine	I	126·85	Thorium	Th	232·5
Iridium	Ir	193	Tin (stannum)	Sn	118·5
Iron (ferrum)	Fe	56	Titanium	Ti	48·1
Lanthanum	La	138	Tungsten (wolframium)	W	184
Lead (plumbum)	Pb	206·9	Uranium	U	239·5
Lithium	Li	7	Vanadium	V	51·2
Magnesium	Mg	24·36	Ytterbium	Yb	173
Manganese	Mn	55	Yttrium	Y	89
Mercury (hydrargyrum)	Hg	200·3	Zinc	Zn	65·4
Molybdenum	Mo	96	Zirconium	Zr	90·7

541. TABLE OF CHEMICAL EQUIVALENTS OF ELEMENTS.

[Hydrogen = Unity.]

Name.	Valency.	Equivalent.	Name.	Valency.	Equivalent.
Aluminium	Al'''	9	Lead	Pb''	103·5
Antimony	Sb'''	40	Magnesium	Mg''	12·2
Arsenic	As'''	25	Mercury	Hg''	100·1
Barium	Ba''	68·7	,,	Hg'	200·3
Bismuth	Bi''	69·5	Nickel	Ni'	29·3
Bromine	Br'	80	Nitrogen	N'''	4·7
Cadmium	Cd''	56·2	,,	N^{v}	2·8
Calcium	Ca''	20	Oxygen	O''	8
Carbon	C^{iv}	3	Platinum	Pt^{iv}	48·7
Chlorine	Cl'	35·45	Potassium	K'	39·15
Copper	Cu''	31·8	Silver	Ag'	107·9
Fluorine	F'	19	Sodium	Na'	23
Gold	Au'''	65·7	Strontium	Sr''	43·8
Hydrogen	H'	1	Sulphur	S''	16
Iodine	I'	126·85	Tin	Sn''	59·2
Iron	Fe''	28	,,	Sn^{iv}	29·6
,,	Fe'''	18·7	Zinc	Zn''	32·7

Note.—The Atomic Weights given on the preceding page are those issued for the current year by the Atomic Weights Committee of the German Chemical Society.

542. EQUIVALENT WEIGHTS OF ACIDS AND ALKALIS.

TABLE I.—WEIGHT OF ALKALI, WHICH NEUTRALISES, OR IS EQUIVALENT TO, UNIT WEIGHT OF ACID.

Acid.	Potassium Hydrate.	Sodium Hydrate.	Sodium Bicarbonate.	Borax.
Sulphuric acid .	1·14	0·82	1·71	3·90
Hydrochloric acid	1·53	1·10	2·30	5·23
Nitric acid . .	0·89	0·63	1·33	3·03

TABLE II.—WEIGHT OF ACID, WHICH NEUTRALISES, OR IS EQUIVALENT TO, UNIT WEIGHT OF ALKALI.

Alkali.	Sulphuric Acid.	Hydrochloric Acid.	Nitric Acid.
Potassium hydrate. .	0·87	0·65	1·12
Sodium hydrate . .	1·23	0·91	1·58
Sodium hydrogen carbonate	0·58	0·43	0·75
Borax	0·25	0·19	0·33

THERMOMETRIC SCALES.

543. There are two different thermometric scales in use in this country, the Centigrade and the Fahrenheit; the former of these is rapidly becoming universal for scientific purposes. The two scales are mutually convertible by the following formulæ, in which F.° represents a temperature on the Fahrenheit scale, and C.° a temperature on the Centigrade scale:—

$$F.^\circ = \frac{9\,C.^\circ}{5} + 32$$

$$C.^\circ = \frac{5\,(F.^\circ - 32)}{9}$$

The temperatures referred to in this treatise are given on the Centigrade scale.

WEIGHTS AND MEASURES.

544. The corresponding values of the French and English weights and measures are here given. The use of the French or decimal system is strongly recommended by its extreme simplicity. The smaller denominations are obtained by taking a tenth, hundredth, thousandth, &c., of the unit chosen; they are designated by the Latin prefixes deci-, centi-, milli-, &c. The higher denominations are 10 times, 100 times, 1000, &c., times the unit, and are named by the Greek prefixes deca-, hecto-, kilo-, &c. Illustrations of this system will be found in the Tables given below.

The starting-point of the French system is the "metre" (=39·37 inches); this is the "unit of length." The "unit of volume" is the "litre," which is 1 cubic decimetre. The "unit of weight" is the gram, which is the weight of 1 cubic centimetre of distilled water at 4° C.

The chief conveniences arising from the use of this system are:—

1st. That all the different denominations can be written as one; since they are either multiples by ten, or are decimal fractions, of the unit. Thus 5 decagrams, 3 grams, 4 decigrams, 8 milligrams would be written 53·408 grams.

2nd. That since 1 cubic centimetre of water at 4° C. weighs 1 gram, we may obtain the *weight* of water from its *measure*, by simply converting the measure into cubic centimetres. The number thus obtained will represent at once the corresponding weight of water in grams. Of course this conversion is strictly accurate only when the water is measured at 4° C. But for ordinary purposes the error introduced, when the water is at the temperature of the air, is too small to be of any importance in the preparation of solutions.

The weights and measures most frequently used for chemical purposes are the gram, the millimetre (mm.), the litre, and the cubic centimetre (c.c.), which is $\frac{1}{1000}$ of a litre.

545. ENGLISH WEIGHTS AND MEASURES.

APOTHECARIES WEIGHT.

lb.		oz.		drms.		scruples.		grains.
1	=	12	=	96	=	288	=	5760
		1	=	8	=	24	=	480
				1	=	3	=	60
							=	20

AVOIRDUPOIS WEIGHT.

lb.		oz.		drms.		grains.
1	=	16	=	256	=	7000
		1	=	16	=	437·5
				1	=	27·343

IMPERIAL MEASURE.

gallon.		pints.		fluid oz.		fluid drms.
1	=	8	=	160	=	1280
		1	=	20	=	160
				1	=	8

1 gallon = 70,000 grains of water at 16°·7 C.
1 fluid ounce = $\frac{1}{20}$ pint = 437·5 „ „ „
1 gallon = 277·280 cubic inches.
1 fluid ounce = 1·733 „

546. FRENCH WEIGHTS AND MEASURES.

MEASURES OF LENGTH.					ENGLISH.		
	metre.	inches.	mile.	furlong.	yards.	feet.	inches
Millimetre =	0·001 =	0·03937 =	.	.	.	.	0·03937
Centimetre =	0·01 =	0·39371 =	.	.	.	.	0·39371
Decimetre =	0·1 =	3·93708 =	.	.	.	.	3·9371
Metre =	1·0 =	39·37079 =	.	.	.	3	3·371
Decametre =	10·0 =	393·70790 =	.	.	10	2	9·7
Hectometre =	100·0 =	3937·07900 =	.	.	109	1	1
Kilometre =	1000·0 =	39370·79000 =	.	4	213	4	10·2
Myriometre =	10000·0 =	393707·90000 =	6	1	156	0	6

1 inch = 0·0254 metre = 2·5399 centimetres.
1 foot = 0·3048 „
1 sq. inch = 6·4514 sq. centimetres.

MEASURES OF CAPACITY.

1 litre = 1 cubic decimetre.

		litre.	cubic inches.	pints.
Millilitre, or Cubic centimetre (c.c.)	=	0·001	= 0·06103	= 0·00176
Centilitre	=	0·01	= 0·61027	= 0·01761
Decilitre	=	0·1	= 6·1027	= 0·17608
Litre	=	1·0	= 61·027	= 1·76077
Decalitre	=	10·0	= 610·27	= 17·60773
Hectolitre	=	100·0	= 6102·7	= 176·07734
Kilolitre	=	1000·0	= 61027·0	= 1760·77341
Myriolitre	=	10000·0	= 610270·0	= 17607·73414

1 cubic inch = 0·01639 litre.
1 cubic foot = 28·31531 litres.
1 gallon = 4·54336 „

MEASURES OF WEIGHT.

1 gram = the weight of 1 cubic centimetre (c.c.) of water at 4° C.

	grams.	grains.	Avoirdupois.		
			lbs.	oz.	drms.
Milligram	= 0·001	= 0·01543			
Centigram	= 0·01	= 0·15432			
Decigram	= 0·1	= 1·54323			
Gram	= 1·0	= 15·43235			
Decagram	= 10·0	= 154·32349	= 0	0	5·65
Hectogram	= 100·0	= 1543·23488	= 0	3	8·5
Kilogram	= 1000·0	= 15432·34880	= 2	3	5
Myriogram	= 10000·0	= 154323·48800	= 22	1	2

1 grain = 0·0649 gram.
1 oz. (Troy) = 31·1085 grams.
1 lb. (Avoirdupois) = 453·593 „

547. USEFUL MEMORANDA.

Weight of 1 c.c. of dry hydrogen at 0° C. and 760 mm. = 0·0000899 gram.

Weight of 1 c.c. of dry air at 0° C. and 760 mm. = 0·0012937 gram.

1 gram of hydrogen at 0° C. and 760 mm. measures 11·16 litres.

1 gram of air at 0° C. and 760 mm. measures 0·773 litre.

Specific gravity of hydrogen, air as unit, = 0·0693.

Specific gravity of air, hydrogen as unit, = 14·43.

Weight of 1 c.c. of mercury at 0° C. = 13·596 grams.

Coefficient of expansion for gases = $\frac{1}{273}$ = 0·003665.

Average percentage of oxygen in air, free from H_2O and CO_2, by volume, = 20·96; by weight, = 23.

548. LIST OF THE CHEMICAL FORMULÆ OF REAGENTS, WITH THE CORRESPONDING CHEMICAL NAMES.

$PtCl_4$	Platinum chloride.
KHO	Potassium hydrate, or caustic potash.
HCl	Hydrochloric acid, or hydrogen chloride.
C_2H_6O	Alcohol.
$NaH\bar{T}$, or $NaHC_4H_4O_6$	Sodium hydrogen tartrate.
KCl	Potassium chloride.
Na_2HPO_4	Sodium hydrogen phosphate.
$AmHO$, or NH_4OH	Ammonium hydrate.
Am_2CO_3, or $(NH_4)_2CO_3$	Ammonium carbonate.
$AmCl$, or NH_4Cl	Ammonium chloride.
$Co(NO_3)_2$	Cobalt nitrate.
$H\bar{A}$, or $HC_2H_3O_2$	Acetic acid, or hydrogen acetate.
$CaSO_4$	Calcium sulphate.
H_2SO_4	Sulphuric acid, or hydrogen sulphate.
K_2CrO_4	Potassium chromate.
$Am_2C_2O_4$, or $(NH_4)_2C_2O_4$	Ammonium oxalate.
Am_2S, or $(NH_4)_2S$	Ammonium sulphide.
HNO_3	Nitric acid, or hydrogen nitrate.
$KClO_3$	Potassium chlorate.
H_2S	Hydrogen sulphide, or sulphuretted hydrogen.
Na_2CO_3	Sodium carbonate.
KCy, or KCN	Potassium cyanide.
$Na_2B_4O_7$	Borax, or sodium diborate.
K_4FeCy_6, or $K_4FeC_6N_6$.	Potassium ferrocyanide.
K_3FeCy_6, or $K_3FeC_6N_6$	Potassium ferricyanide.
$KCys$, or $KCNS$.	Potassium sulphocyanide.
KNO_3	Potassium nitrate, or nitre.
$Pb\bar{A}_2$, or $Pb(C_2H_3O_2)_2$.	Lead acetate.
$SnCl_2$.	Stannous chloride.
$Am\bar{A}$, or $NH_4(C_2H_3O_2)$	Ammonium acetate.

List of Chemical Formulæ and Names—*continued.*

$H_2\bar{T}$, or $H_2(C_4H_4O_6)$	Tartaric acid, or hydrogen tartrate.
H_2SO_3	Sulphurous acid, or hydrogen sulphite.
$CaCl_2O_2$	Calcium hypochlorite.
$AgNO_3$	Silver nitrate.
$CuSO_4$	Copper sulphate.
$HgCl_2$	Mercuric chloride.
HNO_3+HCl	Mixture of hydrochloric and nitric acids, aqua regia.
$BaCl_2$	Barium chloride.
$Ba(NO_3)_2$	Barium nitrate.
$Ca(OH)_2$	Calcium hydrate, or (in solution) lime-water.
$Ba(OH)_2$	Barium hydrate, or (in solution) baryta-water.
$MgSO_4$	Magnesium sulphate.
$K_2Cr_2O_7$	Potassium dichromate.
KI	Potassium iodide.
$FeSO_4$	Ferrous sulphate.
$Na_2S_2O_3$	Sodium thiosulphate.
MnO_2	Manganese dioxide.
CS_2	Carbon disulphide.
KNO_2	Potassium nitrite.
$K_2Mn_2O_8$	Potassium permanganate.
$FeCl_3$	Ferric chloride.
$Na\bar{A}$, or $Na(C_2H_3O_2)$	Sodium acetate.
$AmHMoO_4$	Ammonium molybdate.
$NaAmHPO_4$, or $NaNH_4HPO_4$	Microcosmic salt.

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